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The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

Volume 71 Number 7

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RECORD GROUP SALES

The 28th annual general meeting of Imperial Chemical Industries Ltd was held on June 16th in London.

Dr. Alexander Fleck, the chairman, in the course of his speech said—

The consolidated sales of the Group, after deducting the value of the sales made by our new subsidiaries, have increased from £282 millions to £327 millions—an increase of £45 millions or 16 per cent over the figure for 1953, itself at that time the highest in our history. The Company's home sales and direct exports each increased by approximately the same percentage.

I should like most specially to draw your attention to the table of price indices in which the changes in home trade prices of the Company's products are compared with other significant indices. I am sure you will agree that the comparison shows the Company's prices in a very favourable light. Since 1938 the cost of our raw materials has risen by 272 per cent and the earnings of our workers have risen by 202 per cent. For the same period the Board of Trade wholesale price index increased by 225 per cent. In comparison with these increases (each of which exceeds 200 per cent) the prices at which ICI sells its products in the home market have risen by only 91 per cent. This, I think you will also agree, shows the remarkable success which has attended the Company's policy of keeping its selling prices as low and as stable as possible. Low and stable prices encourage our customers to use more of our products, and as our products are used by many British industries, we believe that our price policy aids and supports the economy of the country as a whole.

Between 1953 and 1954 the Company's price index for home sales has moved up by one point only, and the increase in the value of our home sales during this period is therefore almost entirely due to increase in volume. The consequential increase in profit for 1954 is also attributable to increase in volume, coupled with improvements in efficiency due to Work Study and other factors.

The Company will continue this price policy and will aim not only at absorbing increased costs but, wherever practicable, at seeing that reductions in costs resulting from improved methods and greater efficiency are reflected in lower prices to its customers. Let me emphasise, however, that the price indices I have quoted are averages, and that it may not be possible in all cases to avoid an increase in price; in fact, I am sure that some readjustments upwards in some of our products will almost be inevitable. This is particularly so where raw materials, fuel and freight represent a large proportion of total costs. We are glad to play our part with others first of all in striving after methods of increasing productivity and then of passing on the results of such efforts. Contributions of this nature will, we hope, come from all branches of British industry. It is by such contributions, widespread as they should be, that each one of us must earnestly hope that effective action will soon be taken to prevent the inflation which is threatening to erode our material standards.

EXPORTS AND OVERSEAS INTERESTS

At £67¼ millions the Company's exports were a record and showed an increase of 16 per cent over the previous year. This increase in the value of exports represents a still greater rise in the volume of exports.

I regard this high export figure as a particularly significant feature of the year's trading, because it has been achieved in the face of keen competition from other major chemical producing countries and in spite of the strong demand for our products from the home market. Indeed, if indirect exports were included, it would be seen that more than 40 per cent of the Company's production now goes abroad.

A number of our overseas companies are now substantial manufacturing entities, and when I tell you that the total turnover of the Company's overseas business (both local manufacture and imports from the United Kingdom) through subsidiary and associated companies is now approaching in value ICI's sales in the United Kingdom, I am sure you will appreciate how important our overseas interests are.

It is worthy of note that the net ICI Ordinary dividend, requiring just under £8 millions, represents rather less than 37 per cent of the net income of the year, £21¾ millions. Furthermore, this Ordinary dividend, requiring £14 millions before deduction of income tax, represents a return of 4·8 per cent gross on an employed capital of £295 millions, which according to the Company's balance sheet is the employed capital belonging to the Ordinary stockholders.

EMPLOYEE RELATIONS

I referred last year to the Employees' Profit-Sharing Scheme then being introduced so as to operate from 1st January 1954. The Board regard it as a valuable development in industrial relations and one means of emphasising the essential unity and partnership of all those who are engaged in the Company's business.

It is a visible emblem of that tripartite partnership which we desire to strengthen by all means in our power, that is to say, between the stockholders who supply the risk capital, the people who manage and produce, and the customers without whose good will our efforts would be useless.

Although the majority of the Company's employees will become stockholders of the Company through the Profit-Sharing Scheme, it is the hope of the Board that many employees may be encouraged to increase their interest in the Company by acquiring further stock from their personal savings. Proposals are therefore under consideration for improving our existing Employees' Share Investment Scheme whereby a limited number of Preference and Ordinary shares would be issued to employees at a discount below market price (instead of being bought on the Stock Exchange as hitherto). The discount would not be large, but we hope that it would prove to be attractive.

CURRENT YEAR'S TRADING

As regards current trading, I am pleased to tell you that in 1955 we have so far experienced a period of great activity. The general level of both home and export sales is well above that of the corresponding period of last year. In fact, I do not think I can do better than repeat the words that I used to the stockholders last year and say that 'So far this year the Company has enjoyed, in common with other industries in this country, a period of expansion, and the prospects are in general good.'

This, of course, assumes that the very serious strikes which so gravely affect our national economy are not allowed to recur and that our united efforts can be successfully directed to minimising, if not eradicating, the deplorable setback which the country as a whole has suffered.

On the other side of the account, I would tell you that the increased level of sales this year, to which I have referred, is due to some extent to production now coming forward from new plants which have recently been completed. Competition, particularly overseas, is strong, but we are confident that, provided our prices do not have to be put up because of rising costs at home, we can meet this competition and make still further progress.

The report was adopted.

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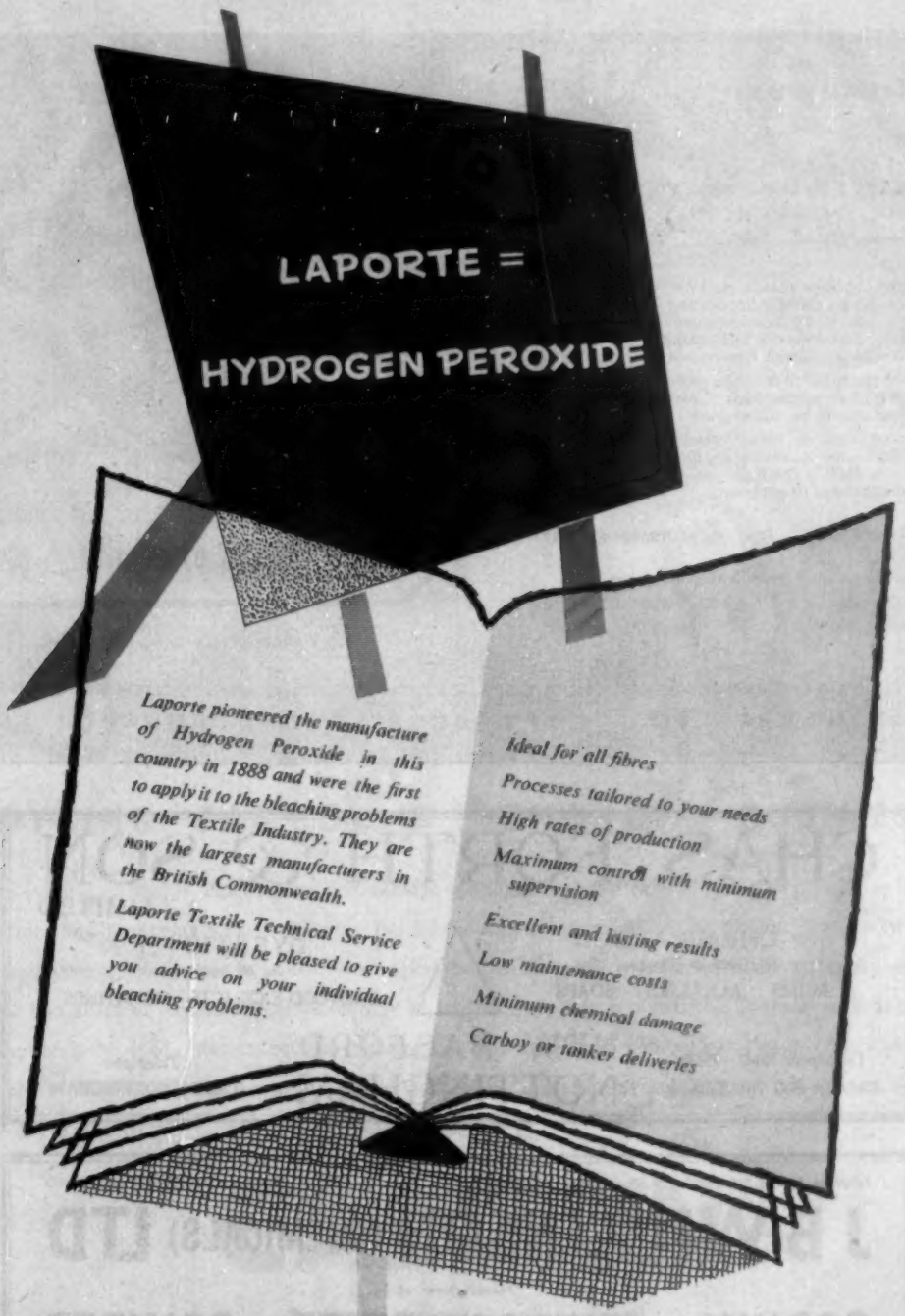

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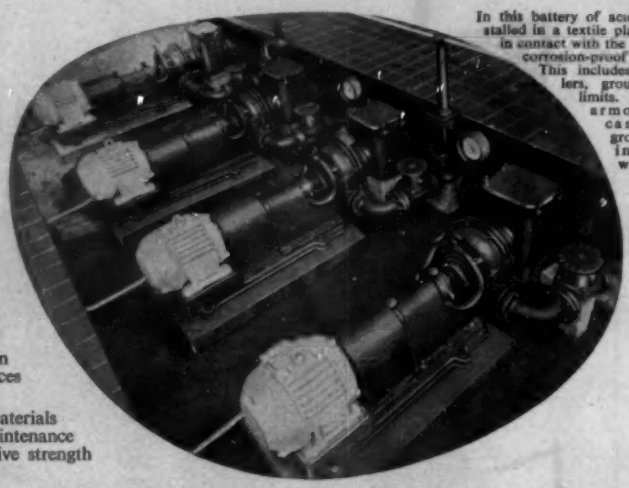

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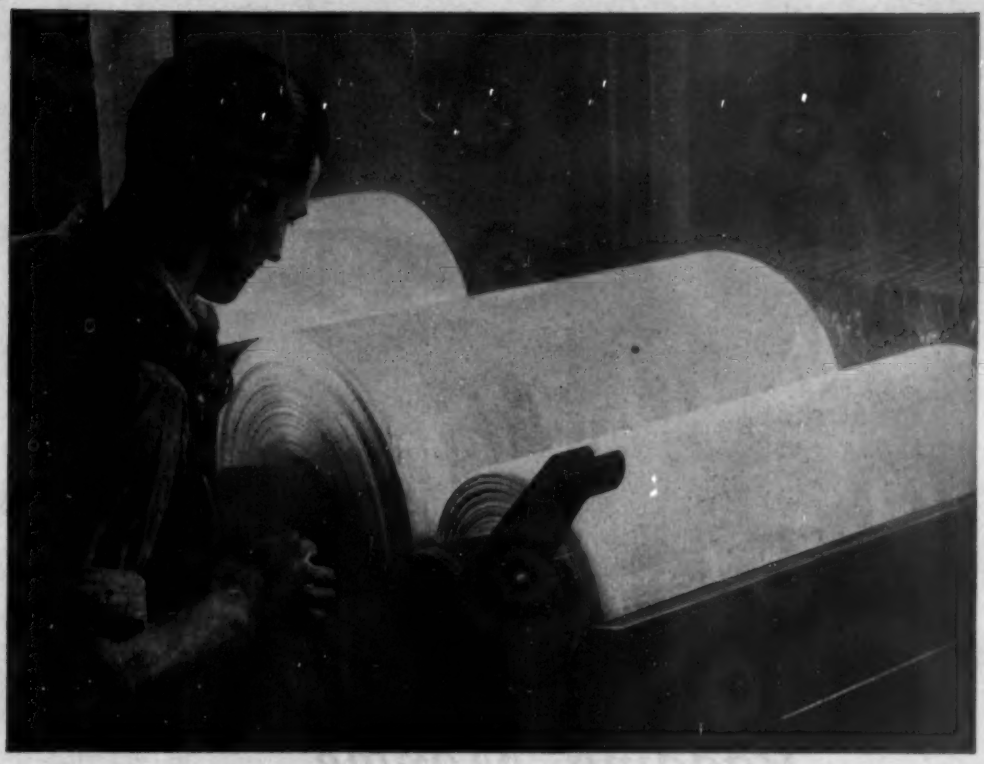

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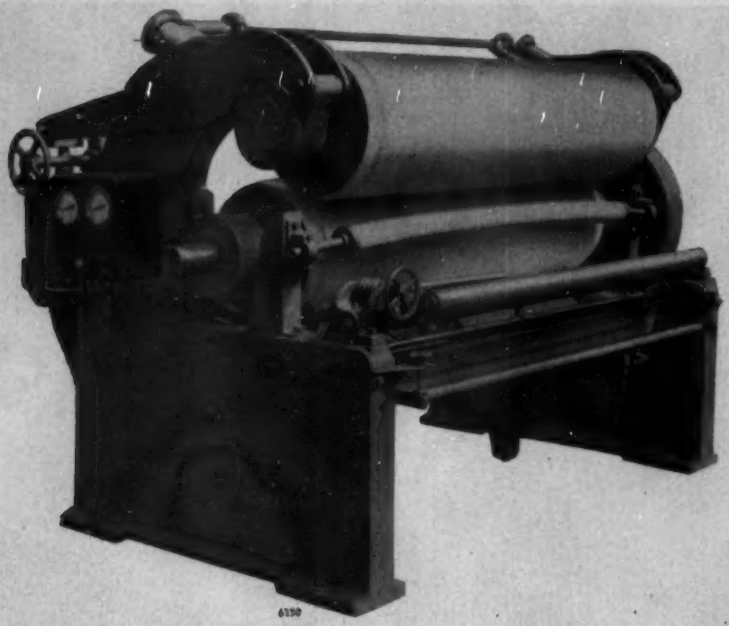

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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Wear Properties of Resin-finished Spun Rayon Fabric—
A New Method of Laboratory Assessment — *L. F. H. Breens and T. H. Morton*

The Dyeing of Terylene Polyester Fibre with Disperse Dyes above 100°c. — *A. S. Fern*

The Dyeing of Textile Fibres at Temperatures above 100°c. — *J. A. Fowler*

The Guilds of the City of London with special reference to the Dyers' Company — *P. E. Jones*

Some Observations on the Dyeing of Wool at High Temperatures — *D. R. Lemin, H. Sagar, and G. A. Coutie*

Cellulose Acetate and Water Repellency — *R. J. Mann*

COMMUNICATIONS

The Polarography of Azo Dyes — *J. de O. Cabral and H. A. Turner*

The Bleaching of Jute with Hydrogen Peroxide — *H. Chatterjee and K. B. Pal*

The Iodination of Wool — *H. R. Richards and J. B. Speakman*

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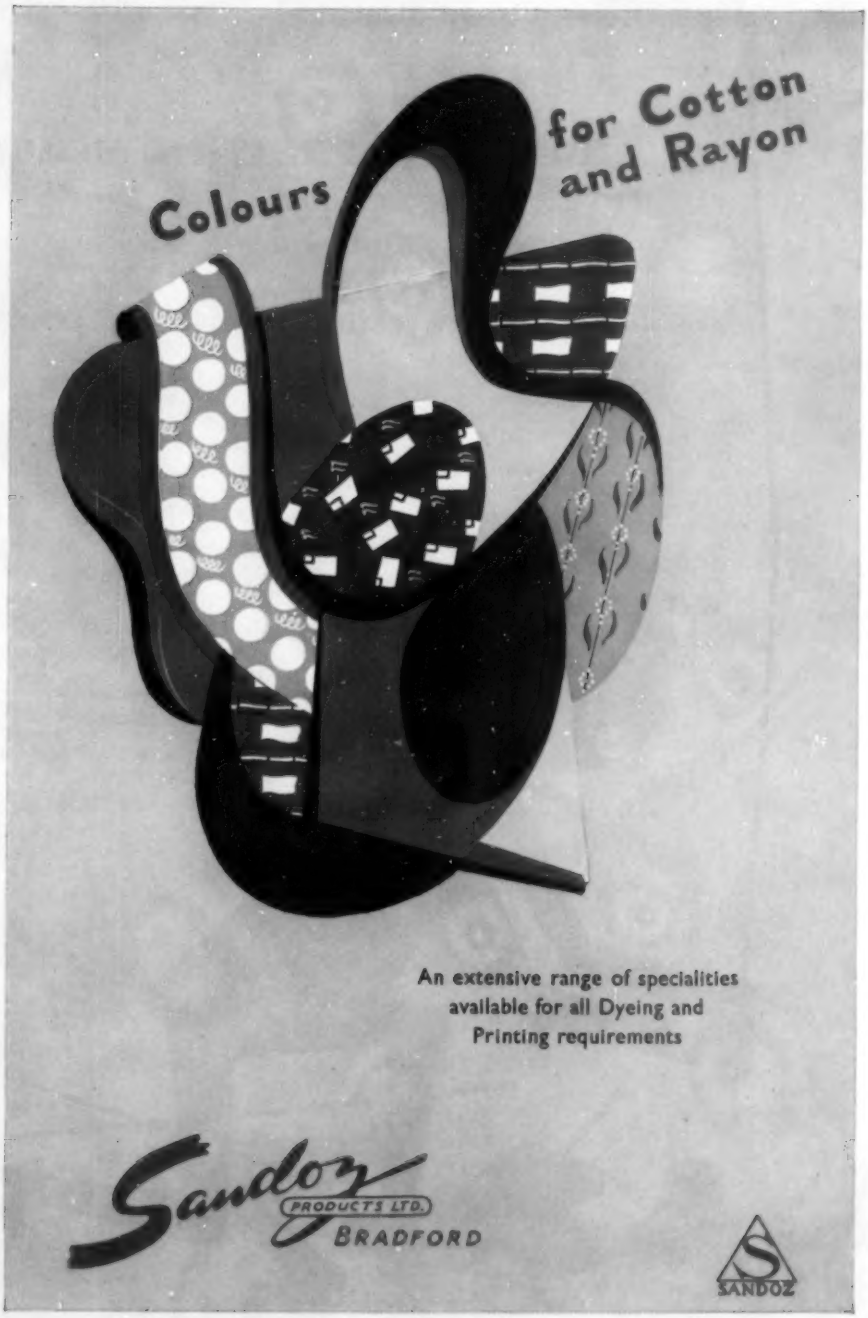
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SECOND LONDON LECTURE

Textile Printing in London and the Home Counties

D. KING

Meeting of the London Section held at the Waldorf Hotel, Aldwych, London, on 5th March 1954, Mr. G. F. Wood in the chair

A historical survey of the development of textile printing in the London area, from the Middle Ages until the present day.

In this paper the history and development of textile printing in the London area is approached purely from the museum point of view. We have, in the Victoria and Albert Museum, a number of textiles stamped with the names of London printers, and we have naturally tried to discover as much as we can about these men and the industry to which they belonged. We very soon found that it was not at all easy to get this information. Although textile printing has been carried on in this country for centuries and has given a livelihood to hundreds of thousands of people, no-one has ever written an adequate historical account of it, and it is only by dint of much rummaging in old directories, Patent Office files, House of Commons reports, registers of births and deaths, and so on that we have managed to collect our facts. We still do not know as much as we would like to know, and the search is still going on, but I think we do now know enough to enable me to give the broad outlines of the history of cloth printing in and around London.

The urge to ornament plain surfaces is a very primitive one. When, many thousands of years ago, long before London existed, men first began to weave cloth, they no doubt decorated it in the simplest and most readily available way, by painting patterns on it with pigments. Examples of such painted cloth have been excavated in Egypt and Russia which are probably more than 4,000 years old, and we can probably assume that in England, too, cloth has been painted since remote antiquity. Julius Caesar tells us that the Britons who opposed his passage of the Thames in 54 B.C. were in the habit of painting their torsos with woad, and we can reasonably suppose that they were perfectly capable of treating cloth in the same way.

But there is no positive reference to the painting of cloth as a London industry until the Middle Ages. There exists today a London livery company known as the Painter-Stainers Company, which is the result of the amalgamation of two of the medieval craft guilds of London, the Painters and the Stainers. So far as I know, the Company no longer has anything to do with cloth printing, but we learn from the guild regulations that originally the Painters were "for painting with colours in oil and size upon timber, stone, iron and such like" and the Staynors "for cloth, silk and such like". The earliest reference to these Stainers, in 1268, is not one that reflects any particular credit on the profession— they were engaged in an extremely violent brawl with the Goldsmiths— but it does show that by the thirteenth century the painting of cloth was an organised London trade. As an example of the sort of work the Stainers did, Fig. 1 shows a burse from the parish church of Hessett in Suffolk, painted with the head of Christ and the symbols of the four Evangelists, dating from the early fifteenth century. From one of the colour recipe-books of that period we learn that these London cloth painters worked in water-colours (made from brazilwood, indigo, verdigris, and copperas) on closely woven linen saturated with gum water, the linen being laid on coarse wool cloths which soaked up the moisture and prevented the colours from spreading. The burse is a small-scale example of their work; they also made large hangings for rooms, painted with quite elaborate scenes, which served as a cheaper substitute for tapestries. Such hangings continued to be made in London until the seventeenth century, and Shakespeare, for instance, makes several references to "painted cloths" of that type.

Printing on cloth, as distinct from painting on cloth, makes a rather late appearance as a London industry. The earliest reference I have found is in the regulations drawn up by the Painter-Stainers Company in 1581, which mention the making of "any kind of painted cloth . . . with patterns, print, stencil or otherwise". Abroad, of course, printing on cloth goes back very much farther than that. There is no evidence, as is sometimes stated, that the Chinese ever used their early printing types for textile decoration, but printed textiles were certainly made in great quantity in Egypt and the Near East all through the Middle Ages, and from the thirteenth century a big European printing industry grew up, centred on the Rhineland, where textile printing preceded and paved the way for printing on paper and printed books. An example of the type of printing done by the Painter-Stainers in 1581 is a fragment of white linen, printed in black, from wood-blocks, with a design imitated from the popular blackwork,

or black silk embroidery of that period (Fig. 2). The whole piece may well have been intended for a cushion cover. Prints of this type continued to be made down to the late seventeenth century. Many of them were, like that in Fig. 2, patterns for or imitations of embroidery. Others were novelties, such as a map on yellow silk printed in black from an engraved plate in exactly the same way as an engraving on paper (Fig. 3); the coloured boundaries are painted in with water-colours. It bears the date 1686 and a dedication to King James II by William Berry at the sign of the Globe between Charing Cross and Whitehall. It is an ancestor of those maps printed on silk which were carried on operational flights during the second World War, and which were worn by women as head-scarves for a year or two after the war. Another kind of seventeenth-century prints were linens with large-scale patterns intended to be stuck on the wall like wallpaper. The example in Fig. 4 shows the heads of a man and a woman, intended perhaps for Charles II and his Queen, and fragments of some classical scene. It, too, is printed in black, and again the colours, which have now almost entirely disappeared, are water-colours painted in with the brush.

Now the sixteenth- and seventeenth-century prints of these types obviously had only a limited sphere of usefulness. The black patterns—printer's ink or lampblack—were reasonably durable, but the water-colours had to be painted in by hand, were often pale to start with, faded rapidly, and, of course, would not stand washing. London printers producing this class of work clearly could not hope to compete for long with the cotton chintzes then beginning to arrive in quantity from India and Persia, with attractive patterns carried out in gay colours which were fast-dyed and could not be washed out. The technique of dyeing patterns into the cloth had probably been carried on in the East for thousands of years, and it is very strange indeed that the secret of it had never before penetrated into Europe. The ancient Romans had at least heard of it. The Roman writer Pliny, who was killed in A.D. 79 through being a little too interested in the eruption of Vesuvius that destroyed Pompeii, included in his great Encyclopaedia of Natural History a description of a dyeing process. He speaks of white cloth being treated in various places with different mordants, and on immersion in a single vat of dye being fast-dyed in several colours according to the properties of the mordants used. It is obvious that Pliny, 1,900 years ago, was giving a quite accurate description of mordant-dyeing with madder, although the precise details of the technique were unknown in Europe and were practised only in Asia. Another technique of dyeing patterns in cloth used in the Near East during the classical period was that of resist dyeing with indigo. On a number of cloths excavated in Egypt which date from around A.D. 400–500 the pattern had been drawn or painted with some sort of wax or clay, and the whole fabric had been dumped in indigo dye liquor. The cloth was dyed blue, with the exception of the parts covered with the wax, and the wax was then boiled off, leaving the design in white.

There were, of course, plenty of highly skilled European dyers, but these processes went on being used in the East for century after century, apparently without ever penetrating into Europe. When in the seventeenth century a lot of Indian calicoes and chintzes began to be imported, they were made by a combination of these two methods — mordant dyeing with madder and resist dyeing with indigo. Actually the whole process of making one of these hangings was very long and complicated, and can be only very briefly outlined here. There was no printing; the whole design was painted by hand, but in fast-dyed colours. The Indian workmen first of all soaked the cloth in a mixture of buffalo's milk and a fruit called *cadou*, which is a myrobalan plum, dried it in the sun, and then beat it with wooden beaters to make it smooth. The design was then drawn in, and the black outlines were made with iron liquor. The next step was the resist dyeing with indigo: the entire cloth except those details which were required to be blue or green was covered with wax, and was immersed in indigo dye liquor, when the dye affected only those parts which were exposed to it and the rest remained white. The wax was then boiled off. The cloth was again soaked in cadou and buffalo's milk, and then the mordants which would produce the reds and violets were painted in; these were various concoctions of which alum was the principal ingredient. The cloth was then immersed in madder dye liquor, which reacted with the several mordants to produce the different shades of red or purple. It was then laid in the sun to bleach the whites, and the last stage was the painting in of a yellow vegetable dye which could also be painted over the blue to give greens. Thus the cloth finished with a dyed pattern in black, blue, two or three reds, two or three violets, yellow, and green on a white ground.

This obviously was something very much better than European printers were producing, and by the middle of the seventeenth century everyone in Western Europe who had enough money to be smart and up-to-date was clamouring for these painted cottons to furnish their houses and make their dresses. The demand was so great that the various East India Companies could not ship them back fast enough. Obviously there was very big money in this for any European who could find out how these things were made and produce a comparable article. With European merchants at large in India, the processes could not remain secret indefinitely, and from about 1675 onwards factories began to spring up, particularly in France, Holland, and England, which claimed to be producing printed stuffs in the Indian fashion. In England between 1675 and 1695 a whole series of patents were taken out by various people for this kind of product. The English factories were all in the London area, and we know the names of most of the factory-owners; some were French and some were Dutch, but many were

English. The industry flourished, and clearly it was also efficient, since very soon the British India Company was writing letters to India complaining that the English prints were selling for less than their Indian counterparts.

The reason, of course, why the English manufacturers could undercut the Indians despite the higher cost of labour in this country was that, while the Indians painted in every detail by hand, the English printed with blocks. They had succeeded in adapting the Indian process of madder dyeing to the established European practice of block-printing. They printed the different mordants on the cloth one by one, and then, by immersing the cloth in the madder dye liquor, the parts so printed took up the various red and violet colours. They did away with the laborious Indian method of resist-dyeing with indigo by devising a form of indigo dye which could be painted directly on to the cloth; the yellow was also painted on as in India. This painting was done by women and girls, whereas the printing and the other operations of the factory were all done by men and boys.

Fig. 5 shows a trade card of one of these seventeenth-century block-printers. He was what was known as a *job-printer*, i.e. he was not attached to a factory but was a free-lance, with his own workshop, prepared to print any cloth that was brought to him for the purpose. His services were frequently used, so we are told, by dishonest servants who stole their masters' linen and then brought it to be printed so that it would not be recognised. There were as many as eighty of these job-printers in London in 1719. The little boy (Fig. 5) is assisting by spreading colour evenly on the sieve; he was then, and still is, called the *tearer*, from the French word *tireur*, and small children were used for this job right up till the nineteenth century. When he has spread the colour evenly, the printer puts his block on it; then he transfers it to the cloth, placing it very carefully so that it joins accurately with the next pattern—there are pins called *pitch-pins* in the corners of the block to help him in this—and gives it one or two sharp blows on the back with his mallet or maul to drive the colour down into the cloth. That, of course, prints only one colour or mordant. He goes right along the whole length of the cloth printing that colour, and then comes back to the beginning, taking a different block and starting to print another colour; and so on until the pattern is complete with all its different colours. This work demands a high degree of skill and experience if it is to be done properly.

The London printworks sprang up in certain well defined localities. Fig. 6 shows the distribution of the most important ones existing in the eighteenth century. Apart from one or two along the Thames banks, most of them were concentrated on the tributary streams of the Lea, the Wandle, and the Cray. One reason for this was that the printworks needed abundant supplies of fresh clear water for their operations, which was not then available in a piped system. Another was that they could harness a certain amount of power from the stream by means of water-wheels. But probably the most important reason for the choice of these particular localities was that the closely related trades of bleaching and dyeing had already been long established in those places. Obviously it was convenient that the printworks should settle down where abundant supplies of bleached cloth were always available—it must be remembered, of course, that all bleaching was done in those days by alternately soaking the cloth in

FIG. 6—Calico Printing Centres around London in the Eighteenth Century

FIG. 1— English Fifteenth-century Burse of Linen, painted in Gold, Silver ,and Colours

FIG. 2— English Sixteenth-century Linen, block-printed in Black

12 in. × 8¼ in.

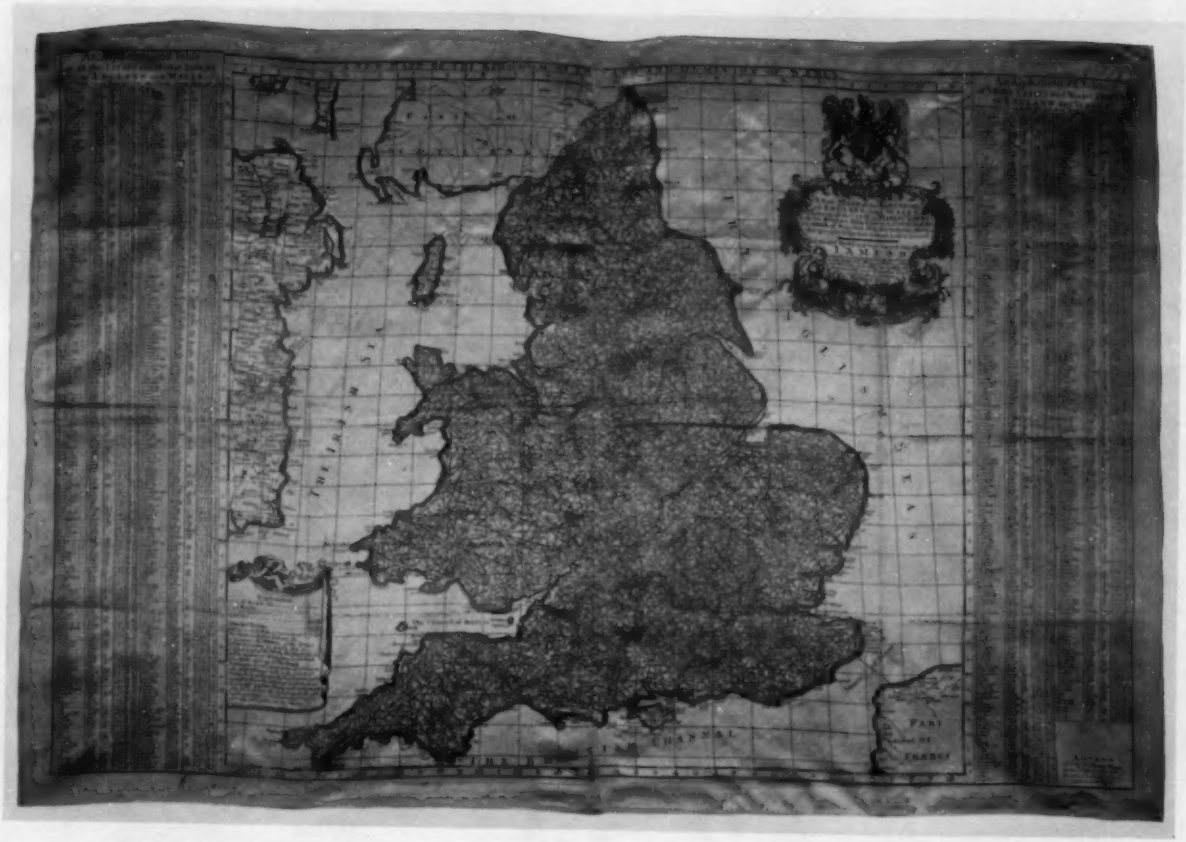

FIG. 3— Map of England printed from Engraved Copperplate on Yellow Satin and hand-tinted in Water Colour, ca. 1686–88
25⅞ in. × 36¾ in.

FIG. 4— Portions of a Seventeenth-century Wall-hanging of Coarse Hand-woven Canvas, printed in Black Outline from Wood-blocks, with Additions of Body Colour put on with the Brush. 37½ in. × 26 in.

Iacob Stampe liuing at ye Sighn of the Callico
Printer in Hounsditch Prints all sorts of
Callicoes Lineings Silkes Stuffs &c
New or Ould at Reasonable Rates

FIG. 5— Trade Card of Seventeenth-century Job-printer

FIG. 7— Block-printed Cotton Coverlet. 120½ in. × 105½ in. George Ormerod, Wallington, Surrey. 1751

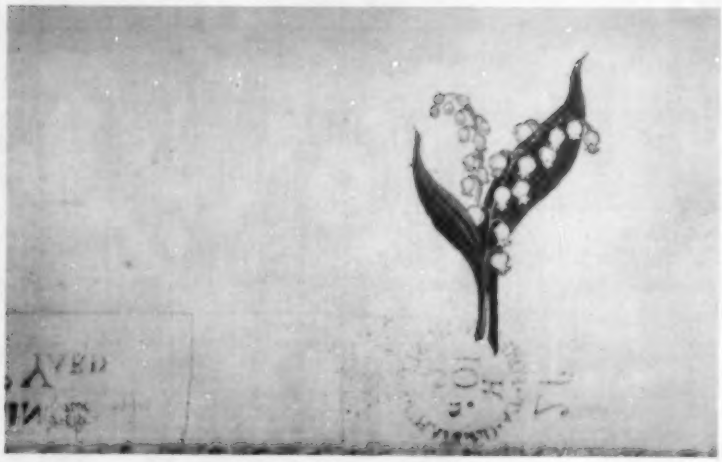

Fig. 8— Late Eighteenth-century Muslin printed by Brown & Co.

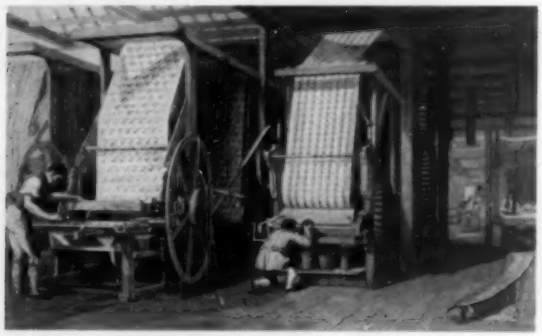

Fig. 9— Printworks about 1830

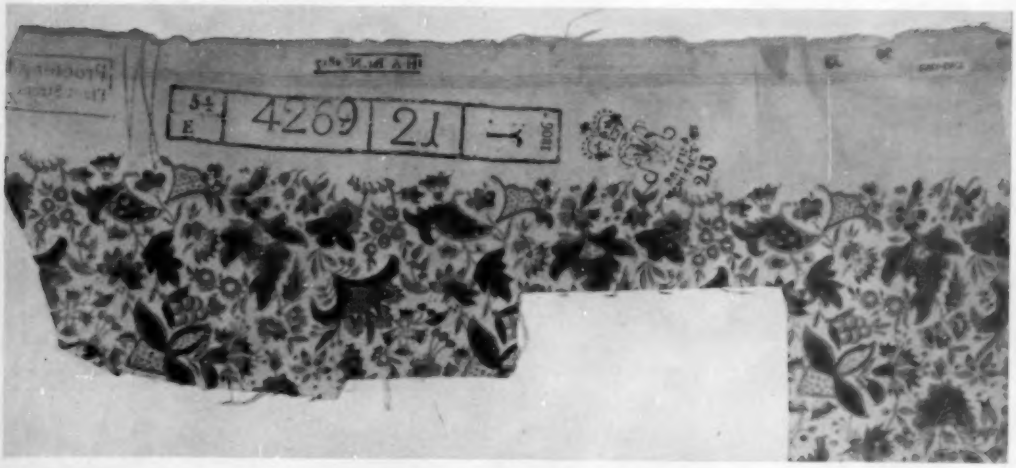

Fig. 14— Block-printed Cotton with Excise Stamp (Early Nineteenth Century)
27½ in. × 16 in.

FIG. 10—Cotton Hanging 118½ in. × 33½ in. printed in Red. R. Jones & Co., Old Ford. 1761

FIG. 11—Cotton and Linen (Fustian) Hanging printed from Engraved Metal, with Superimposed Colour from Wood-blocks. R. Jones & Co., Old Ford. 1769

FIG. 12— Handkerchief printed from an Engraved Plate by Joseph Ware, Crayford, Kent, in the Late Eighteenth Century
30½ in. × 26½ in.

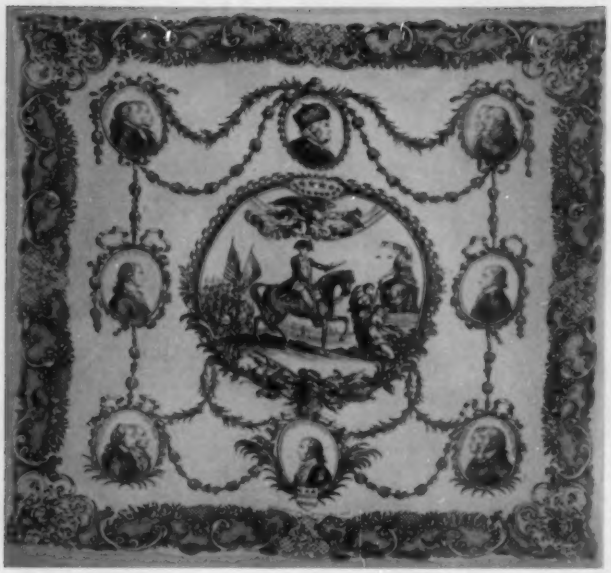

FIG. 13— George Washington Handkerchief, printed by Talwin & Foster, Bromley Hall

FIG. 15— Printed Cotton Panel, "Cray" Pattern designed by William Morris
33 in. × 38½ in.

alkaline lyes and then exposing it on grass, a process which might take up to eight months before the cloth was completely bleached. Again, using the new Indian methods of fast-dyeing patterns, the printworks had need of the skilled dyers who were available in those places; and they needed, too, the dyes which had long been ground in water-mills on these same streams, particularly on the Wandle.

Of individual works, that of William Sherwin seems to have begun operating in 1676, probably at West Ham. In 1696, he stated that his next-door neighbour and himself were employing about 400 hands, which argues a fair degree of prosperity. A few years later, in 1719, the largest factory seems to have been that of the Mauvillon family — evidently French Huguenot refugees — at Mitcham. The list of their staff will give some idea of the organisation of an early eighteenth-century printworks. They employed about 150 hands. Of these 12 were drawers and cutters, i.e. men engaged in the designing and the cutting of the blocks. There were 28 printers, i.e. men who did the actual printing of the cloth. There were 12 grounders, mostly women, who painted in the blue and yellow dyes by hand after the madder-dyeing had been completed. There were 40 tearers, the children who kept the printers and grounders supplied with colour. And finally 60 fielders, who were employed to peg the cloth out in the fields for bleaching and other labouring jobs. The operations of the factory were to some extent seasonal, since in three or four winter months the weather was too bad for this field-bleaching and other outdoor processes.

The prosperity of the trade was given something of a fillip by an Act of Parliament of the year 1700. The English weavers had been claiming for years that they were being ruined by the imports of Indian silks and painted calicoes, and in 1700 Parliament yielded to their clamours and forbade the import of those articles. The weavers, however, soon found that they had done themselves no good at all, since the people who were now unable to buy the Indian prints bought instead, not English wools and silks, but English prints, much to the delight of the printers. So the weavers renewed their agitation — weavers have always been turbulent — and after a number of riotous scenes in which a number of ladies had the embarrassing experience of having their cotton print dresses torn off their backs in the streets, they managed in 1720 to get a law passed which prohibited the use of printed cottons in England altogether. The printers were not, however, very much damaged by this, since they just went on printing on linen, or on fustian, i.e. cloth with a linen warp and a cotton weft, down to 1774, when the great English cotton magnate Richard Arkwright succeeded in getting that prohibition law repealed and enabled Englishwomen once again to wear their favourite cotton prints.

Fig. 7 shows a bedcover or quilt, printed from wood-blocks in several colours, by the firm of George Ormerod, of Wallington in Surrey, in 1751. The derivation in layout and design from the Indian painted hangings is still quite apparent, though the individual motifs are Western in character. Indian styles remained popular also for the smaller dress prints all through the eighteenth century, but the example shown in Fig. 8 is thoroughly English in character, a lily of the valley design printed from wood-blocks in green and yellow on fine white muslin by Brown & Co., who in the second half of the century had their factory at Merton in Surrey. There is in the Victoria and Albert Museum a very pretty complete dress made of that material.

An important innovation introduced in the London factories just after the middle of the eighteenth century was printing from engraved metal plates. As shown in Fig. 3, black prints of this kind had already been made in the seventeenth century. The essential point of the new method was that, instead of printing a black pigment, a mordant was now printed, and the pattern was then fast-dyed with madder dye. At first the pattern was always monochrome, in red or purple, though additional colours could be block-printed or painted in. The printing press used (Fig. 9, left-hand side) was practically identical with the heavy rolling press used in the printing of engravings on paper. The plate was first inked, and then passed through the rollers in contact with the cloth to be printed. This kind of monochrome engraved print has been so universally associated with the Oberkampf factory at Jouy near Paris that it is still known as *toile de Jouy*, but in point of fact it was introduced on this side of the English Channel thirty years before the first one was made at Jouy. The first we hear of such prints is that they were being printed at Drumcondra in Ireland in 1752 by Francis Nixon, who in about 1757 came back to England and started producing them at a factory at Phippsbridge near Mitcham. They immediately had a great success, though they were rather expensive to produce and buy. Benjamin Franklin, who was in London in 1758, bought some to send back to his wife in America.

One of the rare surviving English furnishing prints made by this method, a magnificent engraving of ruins and pastoral subjects printed in red from very large metal plates, is illustrated in Fig. 10. It is marked with the date 1761 and the name Robert Jones, Old Ford, one of the factories on the river Lea. Fig. 11 shows another and in some ways even finer furnishing print from this factory, bearing also the name of Robert Jones but the date 1769. Very gracefully conceived scenes of shooting and fishing are printed in purple from the engraved plate, with the dresses and so on painted in various colours with the brush. Robert Jones's factory was sold in 1780, at which time it had a stock of 200 copperplates, so that obviously at one time prints of this kind must have been quite numerous, although they are so rare today. Jones seems to have had quite a reputation among his fellow calico-printers: Thomas Marlar, who was a cloth

printer at Wallington in Surrey, made a special bequest in his will of 1790 of his "bed, window-curtains and chairs with the covers of Mr. Jones's printing", so that we are probably right in assuming that in prints such as this we see the cream of this particular type of eighteenth-century printing.

The engraved metal plate was used from the 1760s onwards also for the printing of handkerchiefs, the rather large handkerchiefs which are so often seen hanging out of gentlemen's pockets in eighteenth-century pictures, and which were used mainly in connection with the habit of taking snuff. These handkerchiefs were printed with calendars and almanacs, with tables of fares for sedan chairs and hackney carriages, and with all sorts of topical subjects. One example, dated 1769, is interesting as an example of a Masonic handkerchief, bearing various Masonic symbols and the words of a Masonic hymn in the border. It is signed by Mary Ware of Crayford in Kent. Presumably from the same factory and signed by a relative, Joseph Ware, of Crayford, is the handkerchief shown in Fig. 12, printed with a design intended to represent all sorts of papers thrown down pell-mell on a table. There are playing cards, maps, state lottery tickets—the state lottery was a favourite device for raising money for the Exchequer in those days—and various engravings relating to topical events, including one of John Wilkes, who had just won the notorious Brentford by-election. Both of these handkerchiefs are printed in red. There were, of course, a great many other factories turning out this class of goods.

Another similar handkerchief of a few years later is marked with the name Talwin & Foster, Bromley, Middlesex, one of the biggest factories on the River Lea. This establishment had a particularly long history, descending through various members of one family from the 1680s to the 1820s. They were a very prominent Quaker family and probably had many friends and relations in America, which goes some way to explain their printing this handkerchief (Fig. 13) commemorating the American Revolution and glorifying George Washington and other American leaders. It serves as a reminder that many people in this country thoroughly disapproved of the Government's handling of the American colonies and had a very sincere sympathy with the motives behind the Revolution. It is interesting to recall that the first important calico-printing works in America was established in Philadelphia in 1774 by John Hewson, who had formerly been a printer at this Bromley factory. The house in which the proprietors of the Bromley works lived, a fine old seventeenth-century house known as Bromley Hall, can still be seen; it was mentioned several times in Quaker records of the time. One of these narratives records—

> visited . . . at Cousin Talwin's at Bromley, who proposed calling to take us in her coach, but we preferred going in the chaise. Dined and drank tea with them. Walked into the Hothouse and round their garden, a delightful spot. The noble mansion a Palace in miniature. Desired the gardener to see if he could find a bunch of ripe grapes, a cluster of the finest muskodines I ever saw.

It is evident from this that the owner of a printworks in the late eighteenth century could afford to live in some style, and if one looks at maps of about 1800 one notices that, especially round Mitcham and Croydon, which were then of course open country, many of the largest houses with the most extensive estates were occupied by the leading printers. They were certainly among the most prosperous industrialists of the London area, but they were also in a sense having a last fling, since already by then the supremacy in the trade had passed quite definitely to Lancashire.

The Lancashire printworks were new: they began only in the 1760s, but in ten or fifteen years they had already become very serious rivals of the London factories, and by 1800 they were virtually monopolising the market for mass-produced goods of low quality. They had very enterprising directors, and vast amounts of capital were poured into them. They enjoyed a number of natural advantages: the great Lancashire cotton spinning and weaving industry was on their doorsteps; they were able to draw on a great reservoir of cheap unskilled country labour; and coal, also, was close at hand to drive the machines, which from this time onward became very important in the history of textile printing. A machine for printing cloth from engraved metal rollers was patented in London in 1764, but the London printers do not seem to have made much use of it, probably because the results it gave fell far below the standards required for the high-grade work which they were in the habit of doing. The Lancashire manufacturers, on the other hand, were quick to recognise the advantages for their low-grade work of this machine (Fig. 9, right), which would produce as much as fifty or a hundred block printers could do, and by its use they brought about an enormous increase in production. The London works tended to concentrate more and more on the better class of goods, but even here the advantages they possessed were nullified by the low state of commercial morality in the eighteenth century. Competition was of the cut-throat kind, and bankruptcies were very common. The Lancashire firms regularly enticed skilled London block-printers to the north by bribes and offers of high wages, paying them as much as a pound a week, which in those days was reckoned very good pay. The London firms had the best designers, but this did them very little good, since their designs were pirated by the Lancashire factories, often within a few days of their first appearance on the market, and produced by cheaper methods at a very much lower cost. To combat this an Act was passed in Parliament in 1787 which gave a copyright of new designs for two months; the period of this copyright was gradually extended, but nevertheless the stealing of other firms' designs remained a great nuisance in the trade down to the later nineteenth century. Unable to compete with Lancashire in the cheaper lines, some of the London firms turned their energies to very fine and elaborate work, but this was also very costly, with prices up to a guinea a yard, an enormous price for that time, so that they were tending to price themselves out

of the market. Furthermore, they cut themselves off from the enormous export market which the cheap Lancashire prints were now beginning to exploit. In short, the London manufacturers found themselves in a cleft stick, and by the early years of the nineteenth century they had ceased to compete seriously with the Lancashire production.

One burden that weighed on London and Lancashire alike was the excise duty which was imposed on printed stuffs for over a century. In 1712 the Exchequer was looking round for ways of raising money to pay for the War of the Spanish Succession—the one in which we got hold of Gibraltar—and realising that this rising industry was likely to be profitable, they clapped a duty of 3d. per yard on printed cottons. Two years later, in 1714, this duty was doubled, and further additions were made to it at the time when money was being sought to pay for the struggle against the American revolutionaries. The payment of this duty was meticulously enforced on the manufacturers. Each piece of cloth entering each factory was checked and marked individually by a revenue inspector, who stamped it (Fig. 14) with various serial numbers, the date, and so on. Then, when the piece had been printed, the inspector visited the factory again to collect the duty and to affix the duty-paid stamp. Without this second stamp, the printed cloth could not be sold, and the forging of such a stamp was an offence punishable by death. I have never come across any case of a calico printer actually being hanged for this offence, but several of them were detected in the act of defrauding the revenue, and had to disappear hurriedly. With the enormous expansion of the Lancashire industry the enforcement of this tax became uneconomic, and it was finally repealed in 1831, giving still further impetus to the Lancashire factories. The effect of the repeal of the duty from the consumer's point of view may be seen from this quotation written just afterwards: "a printed dress of good materials and a neat pattern, with fast colour, may now be bought for two shillings"; before the repeal of the duty it would have cost nearly four shillings.

This repeal of the duty, however, brought very little benefit to the London industry, and by 1830 London had ceased to be of any real account as a centre for the production of printed cottons. Most of the surviving London printworks had turned over to block-printing of silks and woollens. The fashion for large printed handkerchiefs continued. These were now generally printed on silk with commemorative or political designs, e.g. one printed in 1809 to commemorate the Golden Jubilee of King George III, and objects of this kind continued to be made in London throughout the nineteenth century. With this type of production some of the works succeeded in maintaining a fairly respectable level of prosperity, but many of them disappeared altogether in this period. The last of the considerable factories on the River Lea were removed to Surrey, to Merton, in the 1830s and 1840s. One of these, the works of the Littler family, which had begun at Waltham Abbey in the 1750s, continued operating at Merton down to 30–40 years ago, when it was taken over by Liberty's.

Probably the most important of the London works in the first half of the nineteenth century was that of Charles Swaisland at Crayford—quite a large factory, employing about 500 people, who were employed mostly in the block-printing of silks and woollens. Swaisland specialised in printing Kashmir shawls and other articles of that type; he died in 1865, and his works were sold up. Some of his old printing blocks were bought by David Evans & Co., who still have their printworks at Crayford, one of the oldest firms in the business. And part of Swaisland's works were bought a little later by G. P. & J. Baker, who still carry on the business there. Not far from Crayford, at Dartford, a new printworks was built in 1865, which has since passed through a number of different hands, but that, too, still exists as the Dartford Print Works, now under the control of Messrs. Warner & Sons Ltd.

So much for the later history of the works in Essex—where the industry came to an end in the middle of the nineteenth century—and in Kent—where it is still flourishing after a history of over 200 years. In Surrey, also, textile printing has an unbroken history. Apart from the Littler factory now operated by Liberty's (see above), Merton Abbey is also famous as the home of the furnishing prints which William Morris began producing there in 1883, of which Fig. 15 shows an example. These designs of the Morris factory were a deliberate break with current standards and traditions in both design and execution. They broke away from the extremely naturalistic floral-patterned chintzes, which were then so popular, to a much more formal and strongly organised type of design. They broke away also from the current range of synthetic dyes, and used instead only the soundest and richest-looking natural dyes. For his hand-block printers Morris drew from the reservoir of such skilled craftsmen as still existed in Surrey. The results of Morris's efforts, though perhaps not entirely congenial to present-day decorative tastes, do still set a standard of integrity in style and conscientiousness in execution.

Modern developments of printing—screen-printing and the like—have been introduced into the factories of the London area, though the old traditions of handblock-printing still continue to flourish. The London section of the industry, in fact, which in the first half of the nineteenth century seemed to be in a rapid decline, has held its own remarkably over the last hundred years, and has even shown some tendency to expand. Printed textiles are primarily utilitarian and decorative, and the aim of the industry is presumably to make its products as useful and as beautiful as possible within reasonable limits of cost. But, however useful and beautiful they may be, they are also ephemeral. They get worn out

and they are thrown away—all except some few which it is our duty at the Museum to rescue and preserve from the general oblivion. But, however short-lived they may be, the industry itself preserves a continuous tradition. The people who have been printing cloth at Crayford or at Merton today derive their skill and knowledge from a tradition which is already centuries old in those very places. For hundreds of years there cloth printers have been inventing new technical processes, evolving new techniques, coping with heavy taxation, worried by Government interference. In one point things have improved: if we are foolish enough to defraud the tax collector, we do not run the risk of being hanged for it as our eighteenth-century predecessors did. For the rest, *plus ça change, plus c'est la même chose*. And it is a good thing for the pride and self-respect of the industry to remember its debt to the many generations of predecessors who stand behind us.

DEPARTMENT OF TEXTILES
VICTORIA AND ALBERT MUSEUM
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The Fixation of Direct Cotton Dyes

R. GILL

Meeting of the West Riding Section held at the Victoria Hotel, Bradford, on 25th November 1954, Mr. G. B. Angus in the chair

The various types of compounds used in the fixation of direct cotton dyes on cellulosic fibres are classified according to constitution, and examples are quoted to show the general structural requirements for fixation. A theory is presented to account for the fact that, besides increasing the fastness to wet processing, fixatives in general also may cause change in colour and decreased light fastness.

Unlike the older idea of precipitation within the fibre, this theory enables the effect of the fixative on the dichroism of the dyed fibres to be predicted. Results are presented to show that the resultant effects are as expected, and finally a comparison is drawn between direct dyeings aftertreated with cationic fixatives and acid dyes on wool. The conclusion is drawn that there is a definite limit to the increase in fastness to washing with soap, or other anionic detergents, which may be attained with cationic fixatives generally.

The term *direct cotton dyes* represents a fairly wide range of types of azo dyes solubilised by sulphonic acid or, to a much lesser extent, carboxyl groups. There is available for the dyer a wide choice of colours and general fastness properties, and the more recent developments have led to dyes of high light fastness, approaching that of vat dyes. Compared with vat and azoic dyes, the direct dyes may be applied to cellulosic fibres by simple means and at low cost, but on the whole, unfortunately, the fastness to wet processing generally, and washing in particular, leaves a good deal to be desired.

Various treatments are known, and used, which lead to increased wet fastness in the case of a few selected dyes. For example, some dyes are improved in wet fastness by aftertreatment with formaldehyde; in other cases, where the appropriate groupings are present in the molecule for complex formation, improvement is obtained by aftercoppering or chroming. Again, certain dyes have available in the molecule amino groups capable of being diazotised and subsequently coupled with 2-naphthol or other developer.

However, a demand exists for products which are capable of application by a simple aftertreatment to the majority of direct dyes and which, without affecting the handle of the fabric, will have the following properties—

(1) They will give a substantial increase in the wet fastness of the direct dyeing

(2) They will not affect the colour of the dyeing

(3) They will not decrease the light fastness of the dyeing.

In the past twenty or thirty years a large number of products have been marketed which are readily applied and have no adverse effect on handle, but which, whilst meeting the above three conditions in varying degrees, have their limitations.

Broadly speaking, the fixation products are cation-active, the classical representation of their effect on the dye being—

$$C^+ + D^- \rightarrow CD$$

They may be classified as follows—

(1) SURFACE-ACTIVE CATIONIC MATERIALS—These normally give excellent fastness to water, acid, and alkali, but no improvement, or very little, in the fastness to washing with soap, coupled in many cases with a very marked effect on colour and a decrease in light fastness.

(2) NON-SURFACE-ACTIVE QUATERNARY AMMONIUM COMPOUNDS—This is a rather miscellaneous collection constitutionally, but in effect these compounds are analogous to the first type, generally giving enhanced fastness to washing and in many cases having less effect on colour and light fastness.

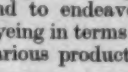
(3) SYNTHETIC RESINS of a number of types, but having some features in common, particularly with regard to the basicity of the various groupings. This group has virtually ousted the two earlier types of fixative, and most commercial materials used today fall into this classification. Here the

fastness to alkali and water is somewhat below that obtained with the surface-active cationics, but the fastness to washing may be quite good and the change in colour very slight. The decrease in light fastness varies amongst the individuals in this class, but the decrease may be reduced to a large extent with some members by treatment in the presence of copper salts.

The next steps are to examine in some detail the products typical of these classes of fixatives and to endeavour to explain the effects on the dyeing in terms of the molecular constitution of the various products.

SURFACE-ACTIVE CATIONIC PRODUCTS

These are products characterised by a molecular structure which can be regarded essentially as consisting of two parts—a long aliphatic chain and a cationic grouping. Typical products[1] are—



$C_{17}H_{33}\cdot CO\cdot NH\cdot CH_2\cdot CH_2\cdot \overset{+}{N}(C_2H_5)_3$

Fixanol C (ICI) Sapamine KW (CAC)

The associated anions are not given in the formulae, since they play no part in the properties to be considered. Other derivatives have been patented, such as dodecyl-sulphonium and -phosphonium compounds—

$C_{12}H_{25}\cdot \overset{+}{S}(CH_3)_2$ $C_{12}H_{25}\cdot \overset{+}{P}(CH_3)_3$

Some products of this class are not fully converted to the quaternary state but are acid salts of the free bases. These show the typical reactions of the quaternary ammonium compounds so long as the pH is sufficiently low to ensure ionisation, but they are obviously sensitive to pH and are generally of little value in aqueous solutions of pH 10 and above.

Surface-active cationic products give excellent fastness to water, acids, and alkalis, but in solutions containing surface-active anions, such as soaps, sulphated fatty alcohols, etc., they give virtually no additional fastness as compared with the untreated dyeing. A typical product of this class, the amide obtained by interaction of stearic acid and unsymmetrical diethylethylenediamine, subsequently quaternised by reaction with dimethyl sulphate, was used in obtaining the curves shown in Fig. 1 and 2. In Fig. 1 a 2% dyeing of Fast Red K (Durazol Red 2BS) on cotton was used, aftertreated with varying amounts of this product for 30 min. at 40°C. The curves show the fastness to immersion for 30 min. in various detergent solutions (liquor ratio 30 : 1) at 60°C. It is readily apparent that, whilst excellent fastness can be obtained to various wet treatments, the fastness to washing in soap is not increased. It should also be mentioned that the colour of the aftertreated dyeings is radically altered, and a very substantial decrease in light fastness also occurs. Fig. 2 shows similar results for 2% Chlorazol Sky Blue FF, obtained under identical conditions.

In 1936 Chwala[2] carried out work on the mode of interaction of typical direct dyes and long-chain quaternary ammonium compounds in aqueous

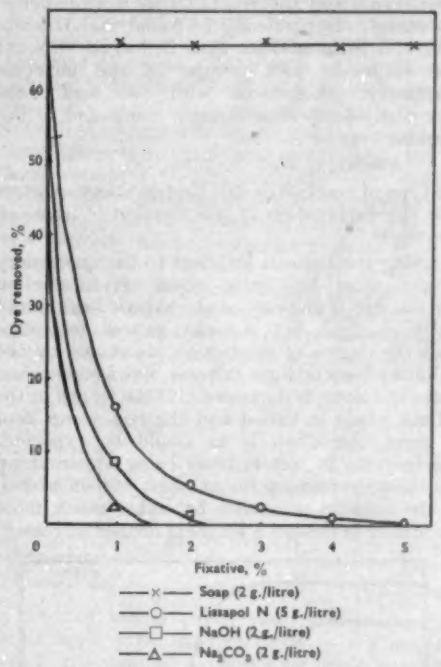


FIG. 1—Fastness to Washing of 2% Fast Red K aftertreated with $C_{17}H_{33}\cdot CO\cdot NH\cdot CH_2\cdot CH_2\cdot \overset{+}{N}(C_2H_5)_2\cdot CH_3$

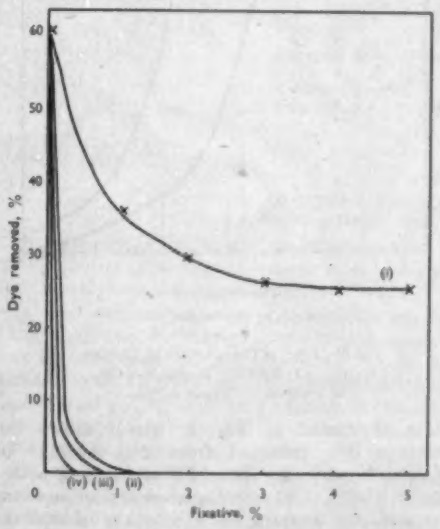


FIG. 2—Fastness to Washing of 2% Sky Blue FF aftertreated with $C_{17}H_{33}\cdot CO\cdot NH\cdot CH_2\cdot CH_2\cdot \overset{+}{N}(C_2H_5)_2\cdot CH_3$

solution, in which he showed that the precipitate formed contained the reactants in stoichiometric proportions. In particular he found that Oxamin Violet and Brilliant Fast Blue B, having two and three sulphonic acid groups in the molecule respectively, precipitated with two and three molar equivalents of a cationic compound of the Sapamine type—

$$D(SO_3^-)_x + xC^+ \rightarrow [D(SO_3^-C^+)_x] \downarrow$$

This type of reaction is still held in many quarters to be the explanation of dye fixation by cationic compounds.

Varying the radicals attached to the quaternary nitrogen atom has little effect on interaction with the dye if the size of the substituents is not greatly changed, but if bulky groups are introduced the degree of interaction, as shown by the resultant effects on light fastness, wet fastness, and change in colour, is decreased. If the length of the aliphatic chain is varied and the end group kept the same, the effect is as would be expected, little increase in wet fastness being apparent up to a chain containing about eight carbon atoms, but the fixation properties becoming much more pronounced as the chain length is further increased.

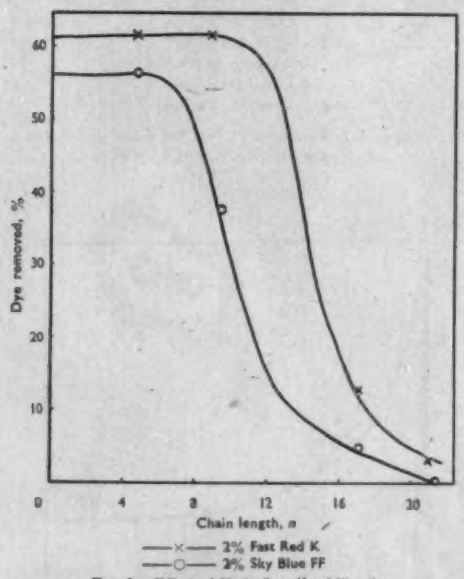


FIG. 3—Effect of Chain Length of Fixative $C_nH_{2n+1}\cdot CO\cdot NH\cdot CH_2\cdot CH_2\cdot \overset{+}{N}(C_2H_5)_2\cdot CH_2$ on Fastness of Dyeings to Soda

This is illustrated in Fig. 3, which shows the percentage dye removed from 2% dyeings of Fast Red K and Sky Blue FF on cotton, after-treated with 1% of a typical quaternary ammonium compound, on washing in a solution of sodium carbonate (2 g./litre) for 30 min. at 60°C.

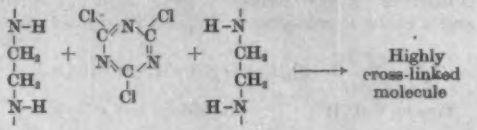
NON-SURFACE-ACTIVE QUATERNARY AMMONIUM COMPOUNDS

The title of this class of fixatives is not strictly accurate, but it is meant to cover basic compounds generally which are not surface-active, other than synthetic resins. This classification is rather wide and covers a large number of patents dealing with very large numbers of possible products, but broadly speaking they are compounds containing two or usually more quaternary ammonium groups in the molecule.

The early patents[3] covered derivatives of the polyethylenepolyamines, the types of compound prepared being quaternised derivatives of general structure—

$$-\overset{+}{N}(CH_3)_2\cdot CH_2\cdot CH_2\cdot \overset{+}{N}(CH_3)_2-$$

The next stage was to prepare more complex polyamines and convert to quaternary ammonium groups. Thus in 1935 I.G. proposed compounds[4] prepared by condensing polyamines and polyhalides, in particular cyanuric chloride—



A little later Sandoz patented[5] similar products from glycerol dichlorohydrins and amines to give an analogous three-dimensional molecule. It is stated in this patent that aftertreated dyeings are improved in wet fastness and washing, generally without modifying the colour of the dyeing or its fastness to light.

It will readily be appreciated that, with polyfunctional reactants in a condensation reaction of this type, whilst the molecular size may be varied by varying the relative amounts of the reactants, the resultant products will have highly cross-linked, three-dimensional structures. In order to obtain a simplified picture of the characteristic molecular organisation of this class as a whole, it is useful to consider the basic structure as a cube, carrying positive charges at the corners—

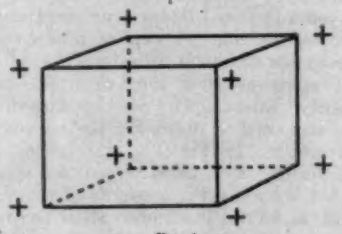
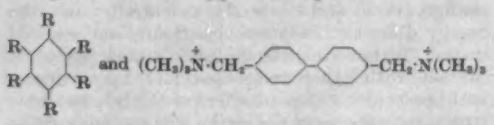


FIG. 4

Whilst a large number of patented fixatives may readily be seen to fall into this simple scheme, there are a number whose structure is not so obvious, but nevertheless they introduce no new features and are merely extensions or limitations of the generalised cubic form. Thus an I.G. patent of 1937[6] claims compounds containing two or more quaternary ammonium groups and no aliphatic chain longer than seven carbon atoms "in order to improve the fastness to washing, which is not obtained by compounds having long aliphatic chains". Appreciable improvement to

washing is claimed, but no mention is made of change in colour. Typical compounds are—



($R = -CH_2 \cdot N^+(CH_3)_3$). It is of interest to note the presence of the diphenyl nucleus in the second molecule, which is apparently designed to be analogous in structure to many direct dyes. So far as the author is aware, these types of compound have not been used commercially.

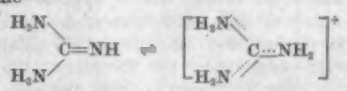
An ICI patent [7] on derivatives of urea which contain quaternary ammonium groups is of value from the theoretical point of view. These compounds may be condensed in the fibre by the use of formaldehyde. Thus it is claimed that padding a direct dyeing with a 1% solution of β-ureidoethylpyridinium chloride, along with formaldehyde and an acid catalyst, and subsequently baking gives very good washing fastness. No mention is made of colour change or effect on light fastness. The interest of this patent lies in the fact that this procedure provides an excellent example of a system in which the simple precipitation mechanism of dye fixation is untenable and one in which the fibre is given a structure analogous to that of acidified protein fibres, i.e. it has positive charges distributed within the polymer structure. Yet the overall results obtained are virtually identical with those obtained by simple aftertreatment with typical commercial fixatives.

RESIN FIXATIVES

The use of formaldehyde resins generally has been known to give, amongst other effects on cellulose, increased wet fastness to direct dyeings. Examples of early patents covering compounds specifically for increasing colour fastness are a Ciba patent [8] of 1934 which covers padding the dyeing in a 15% solution of di(hydroxymethyl)-urea and then baking at 160°C. for 2 min., and a patent taken out by the C.P.A.[9] in 1935 according to which the dyed fabric is padded with dicyandiamide–formaldehyde and then steamed.

A year or two after these, Courtaulds took out a number of patents [10] on reaction products of dicyandiamide, guanidine, etc. with formaldehyde for giving viscose rayon an affinity for acid dyes. These were applied either as a condensate to the viscose mass prior to spinning or as a polymer of low molecular weight, and then baked. A subsequent patent [11], however, covered the preparation of a cyanamide–formaldehyde condensation product which increases the wet fastness generally of direct dyeings by aftertreatment, with little colour change. Most of the products used commercially today have analogies with the subject of this patent (which corresponds to Fibrofix), but from a cursory glance at the patent literature this is rather difficult to see.

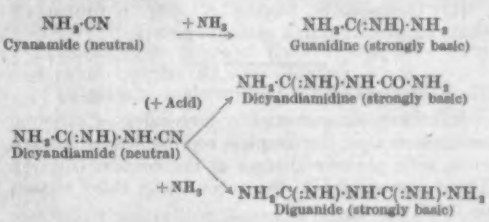
It can readily be appreciated that the action of the two earlier classes of fixation agents is due to e positively charged molecule forming a salt with the dye anion. Since aqueous solutions with which a dyed cellulose material may come into contact in practice can have pH values up to 10 or 11 (in the case of washing with soap or solutions containing sodium carbonate), it is also apparent that only bases which are ionised at least up to these pH values will be of value in commercial use. Thus the simple amines are excluded, and more strongly basic groupings are required. This may be achieved either by quaternising the nitrogen atoms or by utilising groupings in which the cationic form is stabilised relative to the unionised form. The classic example of a molecule in which this latter mechanism is operative is guanidine, in which the addition of a proton gives a cation which is symmetrical and possesses enhanced resonance energy compared with the unionised molecule—



The increased basicity of these modified amine types is as follows—

$R \cdot NH_2$	$NH_2 \cdot C(:NH) \cdot NH_2$	$\overset{+}{N}R^1R^2R^3R^4$
Primary aliphatic amine ($pK = 10{\cdot}5$)	Guanidine ($pK = 13{\cdot}5$)	Quaternary ammonium salt (Fully ionised)

Most of the so-called resin fixatives contain substituted guanidine groupings in the molecule, which are obtained generally by using cyanamide or dicyandiamide as starting materials and forming the strongly basic groups either prior to, or in the course of, condensation with formaldehyde. The following schemes indicate the chemistry of these reactions—



In passing, it is worth mentioning that dicyandiamidine and diguanide are well known to form metal complexes quite readily.

In the simplest case of guanidine, it may be noted that the structure is closely analogous to that of urea, and in fact guanidine reacts with formaldehyde to give highly cross-linked three-dimensional polymers closely related to the urea–formaldehyde condensation products utilised in the crease-resist process.

A Geigy patent [12] of 1946 based on the above reactions claims: "acid soluble condensation products from dicyandiamide, urea, an ammonium salt and formaldehyde, comprising heating together substantially one mol of dicyandiamide with urea and ammonium salt together equal to at most one mol, and condensing the so-obtained reaction product with at least two mols of formaldehyde". A Sandoz patent [13] of 1948 covers water-soluble products obtained by condensing hydroxyalkyl-

guanidines or hydroxyalkyldiguanides with aldehydes. The organic derivatives are not used as such but are obtained in crude mixtures by reactions such as heating together monoethanolamine hydrochloride, dicyandiamide, and formaldehyde for 30 min. at 95°C. In this patent it is stated that these compounds form complexes with various metals and that by the use of metals such as copper, chromium, iron, etc. it is possible to increase the efficiency of the products.

Use of Copper in Aftertreatment with Resin Fixatives

With one or two commercial fixatives either it is recommended that copper salts be used along with the fixative, or alternatively copper is incorporated in the product sold by the manufacturers. Normally the metal derivatives can be applied only to a selected range of dyes, and even in a small range the colour change may be quite large. There are two principal reasons for the use of copper, either to enhance the increase in wet fastness or to decrease the effect on light fastness. Two general mechanisms are possible to give the increased wet fastness: firstly the number of positive centres on the molecule of the fixative may be increased; and secondly, complex formation may occur between the already complexed copper and dye molecules containing suitable groupings. The first mechanism is not of very great importance as a rule, but the second effect may be quite valuable with selected dyes.

In summarising this section, therefore, we may state that the fundamental structural requirements of fixation agents are of two principal types—

(1) Compounds having a long hydrocarbon chain terminating in a cationic group—

These correspond to the first class of fixative.

(2) Three-dimensional molecules carrying cationic groups, the simplest representation being a cube with positive charges at the corners (Fig. 4). These correspond to the second and third classes of fixative.

Theory

ABSORPTION OF LIGHT BY DYES

The absorption of light by dyes has been well treated elsewhere [14], but it is of value to summarise the general position insofar as it is of interest to the present case.

A highly conjugated molecule, typical of dyes, may be considered initially in the hypothetical ideal state of a single molecule isolated in space. On irradiation with white light, energy may be absorbed in specific or quantised amounts and result in the transition of an electron of the π-electron system from the ground state to a higher energy level. Superimposed on this electronic energy absorption are variations in vibrational and, to a lesser extent, rotational energies which result in light energy being absorbed on both sides of the characteristic wavelength corresponding to the pure electronic change.

Thus the light absorbed by an isolated molecule is characteristic of its molecular and electronic configuration and depends essentially on the energy difference between the ground and excited states. If, however, the molecule is now placed in contact with other molecules, and in particular with molecules which interfere with the electronic structure, the energy difference will probably differ from that of the isolated molecule, and hence the wavelength of light absorbed will differ. An obvious extension of this to dyes adsorbed on any given substrate is that, if the dyes are placed in a different environment, e.g. if they are adsorbed on a second substrate, the probability is that the colour will be altered [15].

In order to investigate changes in the fastness of dyeings to light, attention should be directed to the excited state. Considering again the isolated molecule first, we know that this is a state which may have considerable vibrational energy, and this, coupled with the weakening of the valency bonds caused by the electronic transition, often leads, with small molecules, to scission of the molecule, i.e. photodissociation. It is very unlikely, however, that this effect will be apparent with a large conjugated molecule, such as a dye molecule, where excess vibrational energy may be dissipated over the whole structure, and the electron transferred to a different level is not located in a single bond.

On the other hand, dyes of commercial value undergo photochemical change, i.e. fade, only after a large number of transitions (from ground state to excited state and back to ground state), and it is not impossible to imagine that rupture of a valency bond could occur in particularly fortuitous, and therefore improbable, circumstances.

However, on the whole it would seem more reasonable to look more closely at the excited state as a diradical [16] or, more generally, as a chemically highly reactive organic molecule, stable in isolation but liable to undergo reaction if in contact with other molecules, e.g. oxygen, as is the case in any practical system.

In either case, whether fading occurs as a result of rupture or chemical reaction, it is quite clear that destruction or change of the dye molecule would be eliminated entirely if the excited molecule could return immediately to the ground state. For the ground state to be re-attained, the excess electronic energy must be dissipated as heat, and evidence relating to this energy conversion and dissipation is to be found in work on the quenching of fluorescence. It is well known that fluorescence is quenched, for example, by dye aggregation in solution, and in general it would appear that this is due to van der Waals forces, particularly the dispersion forces [17], between the dye molecule and its neighbours.

Thus the conclusion reached is that with any one dye, or with any series of related dyes, the light fastness will vary with the substrate [18], and will tend to be greatest in those systems in which the dye is most strongly adsorbed, by van der Waals forces, to the substrate.

NATURE OF THE DYE–CELLULOSE LINK

A great deal has been written on the nature of the forces which bind the direct dye molecule to a cellulose chain. One conclusion reached, which has been widely held, is that the predominant link is due to hydrogen bonding, but while an appreciable amount of information has been gathered in support of the hydrogen bond theory, there have always been certain points which are very difficult to reconcile with this idea. Foremost among these is the fact that water itself forms hydrogen bonds with molecules containing either donor or acceptor groups for protons, and therefore, for any single hydrogen bond to be formed between cellulose and dye, two hydrogen bonds would have to be broken, between cellulose and water and between dye and water. Furthermore, whilst intramolecular hydrogen bonds are normally maintained on dissolution in water, intermolecular hydrogen bonds are normally broken, a simple example being the equilibrium between carboxylic acids in the monomeric form in water and in the hydrogen-bonded, dimeric form in non-polar solvents. Other points against hydrogen bonding are outlined by Robinson [19], and in this paper good evidence is put forward on steric grounds for the alternative theory of van der Waals forces as being the principal cause of the attachment of direct dyes to cellulose.

It would seem more reasonable, therefore, to ascribe substantivity to generalised adsorption forces rather than to hydrogen bonds, and if this is the case we should be able to apply the conclusions regarding change in colour and decrease in light fastness arrived at in the preceding section. Thus, if on treatment with a fixation agent a direct dye is removed from the cellulose molecule and precipitated within the fibre, we would expect in general that the colour would be changed and the light fastness decreased.

With regard to the change in colour, it is not really possible to predict whether the absorption spectrum of the dyeing will be shifted towards longer or shorter wavelengths. One might expect, from the known effect of van der Waals forces in shifting absorption to longer wavelengths, that, by removal of a dye from its strongly adsorbed state on the cellulose, aftertreatment would shift absorption to shorter wavelengths, but this ignores steric and other effects.

On the other hand, with regard to light fastness, it is possible to state that this will not increase but, if changed at all by aftertreatment, will decrease. This point is particularly noteworthy, since cationic softeners in general are well known to affect the light fastness of direct dyes adversely. It should be pointed out here that there is another possible mode of action of fixation agents, or other products, in decreasing the light fastness of dyes. This is that the action of light on the product itself may give rise to chemically active entities, e.g. free radicals, which in turn react with the dye molecule. This effect is not general, however, since it is difficult to see how a simple quaternary ammonium compound such as cetyltrimethylammonium bromide will be affected by light, but it may account for the action of one or two resin-type products, with which the decrease in light fastness may be counteracted by the formation of metal complexes.

DICHROISM

If the simple precipitation theory of dye fixation is the correct one, a simple test is available in dichroism, and since we should have a system of dye on cellulose which is initially strongly dichroic [20] and finish up with a random precipitation which would show no dichroism, we should also be able to assess qualitatively the extent to which precipitation has occurred within the fibre.

In order to examine this point, a number of direct dyes were examined on viscose rayon initially, after treatment with a fairly typical example of the resin class of fixative (a cyanamide–formaldehyde product as Example 1 of *BP* 515,847 [11]), and after treatment with a typical long-chain quaternary ammonium compound. The conditions of application were rigorous, involving treatment with the fixative at 80°C. for 30 min. at a 30 : 1 liquor ratio, in order to achieve equilibrium distribution.

The results are given in Table I, and from these and other observations the following conclusions may be drawn—

(1) The quaternary ammonium compound leads to virtually complete random precipitation of the dye inside the fibre.

(2) The resin-type product, which has much less effect on colour generally than the surface-active type, has correspondingly less effect on the dichroism.

(3) On aftertreatment with the surface-active quaternary ammonium compound, the change in colour is exactly the same as that obtained by adding excess of the product to an aqueous solution of the dye.

TABLE I

Dye (2% on viscose rayon)	Change in Colour		Dichroism		
	2% Product A*	2% Product B†	Untreated	2% Product A*	2% Product B†
Diazol Light Violet 3R (Fran)	Little change	Much redder	Very strong	Strong	Very weak
Durazol Fast Blue 2GS (ICI)	Greener	Redder	Very strong	Weak	Weak
Diazol Light Red 6BL (Fran)	Slightly bluer	Redder	Very strong	Strong	Weak
Chlorazol Fast Orange RS (ICI)	Slightly redder	Much yellower	Strong	Strong	Weak
Durazol Red 2BS (ICI)	... Slightly yellower	Much yellower	Very strong	Strong	Nil

* Product A— Cyanamide–formaldehyde condensate as Example 1 of *BP* 515,847 [11].

† Product B— $C_{17}H_{35}\cdot CO\cdot NH\cdot CH_2\cdot CH_2\cdot \overset{+}{N}(C_2H_5)_2\cdot CH_3$.

(4) The effect is progressive; e.g. on treating a dyed fabric with a quaternary ammonium compound, the extent of change in colour is related to the decrease in the dichroism.

(5) The increase in wet fastness is not related to the decrease in dichroism.

The last point may be amplified. If a dyeing is treated at 20°C. with a particular long-chain quaternary ammonium compound, the change in colour and the decrease in dichroism are much less than those obtained by treating at 80°C. for the same period, but the difference in wet fastness is very slight. Similarly, treating a given dyeing with a resin-type product and a quaternary ammonium compound can give the same degree of wet fastness (better to washing with the resin type), but less effect on colour and dichroism in the former case than the latter.

These observations may be compared with some remarks by Ackley[21] in a paper on the effect of cationic finishing agents on direct dyes, where he states: "whilst treatment with ½% of a particular finishing agent at 100°F. for 15 minutes produced no serious effect on light fastness, increasing the temperature of application to 150°F. produced a noticeable decrease in light fastness".

MODE OF FIXATION OF DYE

In order to explain the results noted, the following pictorial representation is helpful.

If we take a dyed cellulose fibre and immerse it in water, the fibre swells and the cellulose chains are allowed much freer movement, which results in holes through which dye or fixative molecules can readily pass. The size of these holes is not fixed, but varies quite considerably with time in the amorphous regions of the cellulose, owing to the movement of the polymer chains. Now in general these polymer chains will behave towards entering cations as chains carrying negative charges distributed along their length, since nearly all the dye molecules, especially at low temperatures and in the case of fast dyes, will be firmly adsorbed on the polymer chains, and only a very small proportion of the total dye on the fibre will be present in solution in the imbibed water. Hence the entering large cations will not, unlike sodium ions, remain in the aqueous phase as a diffuse system, but will be adsorbed on the negative sites, as shown in Fig. 5A (before treatment) and Fig. 5B (after treatment) for fixatives of the surface-active cationic type.

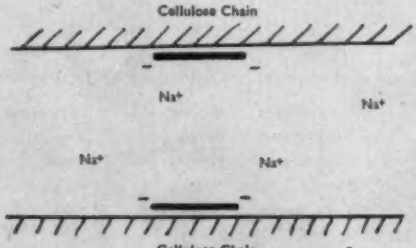


FIG. 5A

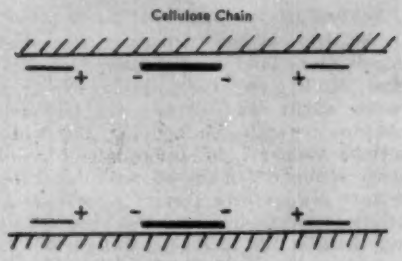


FIG. 5B

This ideal state will result in increased wet fastness, and on the theory outlined above will not affect colour, light fastness, or dichroism. Furthermore, such a system should lead to very rapid penetration of the fibre by a suitable cation, since a concentration gradient is immediately set up which will greatly favour entry of following cations.

The above system, however, is not necessarily in equilibrium, and on increasing the temperature a second state of distribution can be envisaged, resulting in incorporation of the dye in a micelle type of structure with the quaternary ammonium compound, as shown in Fig. 6. This corresponds to the classical method of fixation by precipitation and should result in increased wet fastness, change in colour, and decreased light fastness. Similarly, the dichroism should be nil and the colour of the treated dyeing should be that of a solution of the dye in excess of the quaternary ammonium compound, where the dye will be present in micelles of the same type held in dispersed form by the excess of surface-active agent. It can readily be shown experimentally that this is true.

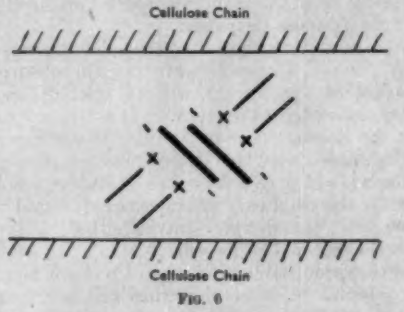


FIG. 6

It will be apparent that the arrangement shown in Fig. 5 corresponds to low-temperature treatment with commercial cationic softeners or surface-active fixatives, whereas Fig. 6 corresponds to high-temperature treatment leading to complete internal precipitation, where this is the most stable state. In the case of the non-surface-active fixatives of the second class and resin-type fixatives of the third class we have a difference, since these will not form micelles. Now the precipitate formed between a three-dimensional cation and a dye in aqueous solution will be due, assuming no penetration of the cation by the dye,

to two principal forces, ionic and van der Waals or adsorption forces. If, however, a further component such as cellulose is introduced into the system, for which the dye has particularly strong adsorption forces, it may well be that the lowest energy state is one in which dye, cellulose, and multivalent cation are so arranged that the dye is adsorbed on the cellulose by adsorption forces and utilises only the ionic forces of the fixative. Such a system corresponds to that shown in Fig. 5B, and this would appear to be the main form of fixation by the resin type of fixative (Fig. 7).

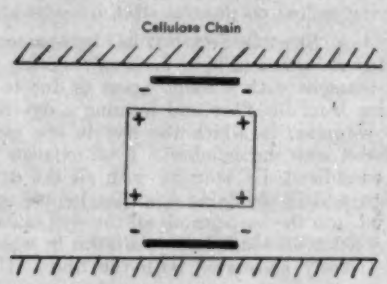


FIG. 7

As stated earlier, this corresponds to an ideal state, and in fact the overall commercial efficiency of a fixation product would correspond to how closely this state is attained in practice.

As an example of the possibility of approach to this ideal behaviour, it may be mentioned that on treating the dyes listed in Table 1 with Fixogene TN Extra Liquid (Fran), the decrease in the dichroism of the treated dyeings is very small indeed, and this is reflected in the slight change in colour and maintenance of the light fastness, whilst the normal increase in wet fastness is obtained.

ANALOGY WITH ACID DYES ON WOOL

A large amount of work has been carried out on the theoretical aspects of wool dyeing, with particular reference to the ionic or coulombic forces between the charged protein fibre and the anion of the dye. If we disregard metal-complex dyes, the three forms of bonding which are possible are coulombic forces, van der Waals forces, and hydrogen bonding. Whilst the first of these has received much attention, there is little doubt that purely ionic bonding alone is very rare, even with the most level-dyeing acid dyes. Since hydrogen bonding is probably of little consequence in aqueous solution, for the reasons referred to in the discussion of cellulose dyeing, we may say generally that a dye is adsorbed on wool by ionic and van der Waals forces combined. The ionic forces are present with all dyes, but the contribution of the van der Waals component varies very widely, from very small in the case of a level-dyeing dye to relatively large in the case of an acid milling dye. Now in the case of dyed cellulose also the van der Waals forces vary widely, from the smallest value in the case of level-dyeing dyes to the largest in the case of dyes of high affinity, and on the theory outlined earlier, aftertreatment with a fixative adds an ionic component. The essential analogy is shown diagrammatically in Fig. 8A and 8B.

FIG. 8A

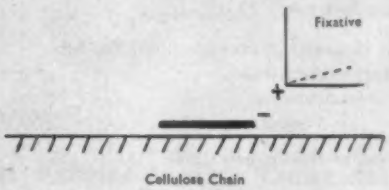


FIG. 8B

It will be appreciated, therefore, that the function of the fixative is to give the additional ionic bonding, and consideration of this point can give an estimate of the maximum efficiency possible with a commercial fixative. In other words, it is possible to state whether it is likely that an aftertreatment of this type can be found which will increase the wet fastness of a dye such as Fast Red K to that typical of vat dyes.

Conclusion

If the postulate is accepted that cationic fixatives owe their effect on wet fastness to ionic forces between the dye anion and the cation, it follows that if the aftertreated dyeing is placed in a solution containing other anions, these will compete with the dye for the ionic sites of the fixative; and if the added anions are successful, the wet fastness of the aftertreated dyeings in the solution will revert to that of the original dyeing. Now simple anions such as chloride or sulphate have little effect, because of behaviour which is analogous to that of acid milling dyes on wool, but long-chain or multivalent anions will have more effect. In a soap solution not only will multivalent anions, in the form of micelles, compete for the fixative, but also, once a fixative molecule becomes attached to one or more soap molecules, it will be removed from the sphere of action by incorporation in soap micelles. Surface-active cationic materials are particularly susceptible to this effect owing to their hydrophobic chain, and hence the fastness to soaping of a dyeing aftertreated with one of these compounds is virtually that of the untreated dyeing. Since the increased fastness to washing of acid milling dyes on wool as compared with level-dyeing acid dyes is due primarily to adsorption forces, and since the adsorption forces of a direct dye on cellulose are uninfluenced by a commercially acceptable fixative (owing to the necessity for avoiding change in colour and decreased light fastness), it follows that the greatest increase

of fastness to washing possible in any given case is relatively small. In support of this conclusion it may be mentioned that comparison of the effects of four commercial fixatives (same weight of 100% fixative) on the washing fastness of dyeings of Fast Red K and Sky Blue FF on cotton showed remarkably little difference in efficiency.

It may be concluded, therefore, that, as regards increasing the wet fastness of direct dyes generally by aftertreatment with cationic compounds, there is little prospect of any further improvement. On the other hand, it may be possible to find new products which will have no effect at all on colour or light fastness.

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Discussion

Mr. A. MURRAY: Products such as Fibrofix often adversely affect the light fastness of direct dyes on cellulosic fibres, and the lecturer has put forward a theory to explain the reason for this effect. Could the same theory or an extension of it explain why copper salts used in conjunction with Fibrofix tend to maintain the original light fastness with many direct dyes?

Dr. GILL: The effect of copper salts in conjunction with Fibrofix may be explained in one of two ways—

(*a*) Fibrofix on exposure to light gives active entities which in turn attack the dye, and this effect is diminished by copper complex formation with the fixative

(*b*) Fibrofix does tend to give a greater decrease in dichroism than some fixatives, and copper complexes may have less tendency to remove the dye from its attachment to the cellulose molecule.

Mr. MURRAY: The lecturer has explained why the products under discussion do not in general greatly improve the fastness to soaping of direct dyes, although they do improve the fastness to other products. Does it follow from the same theory that soap is a better retarding and levelling agent for direct dyes than other products, e.g. sulphated fatty alcohols?

Dr. GILL: The effect of soap on aftertreated dyeings is due to competition with the dye for the positive sites of the fixative. This is therefore not related to the effect of surface-active anionic agents in dyeing, where no positive sites are present.

Mr. J. V. SUMMERSGILL: It has been suggested that any reduction in light fastness resulting from aftertreatment with a fixing agent is due to dye diffusing from the fibre and forming a dye–fixing agent complex, in which the dye is less closely associated with the cellulose. If an extreme case were considered, i.e. starting with all the dye in solution, adding the fixing agent, and entering the material into the suspension, all the dye taken up by the cellulose would be in the form in which it is less closely associated with the fibre. Under these conditions the maximum reduction in light fastness would be expected. Have any experiments on these lines been tried?

Dr. GILL: I agree that in this hypothetical situation maximum decrease in light fastness would be expected; this has not been investigated experimentally.

Mr. R. K. FOURNESS: What is Dr. Gill's explanation of the difference in the effects of soap and alkali on an aftertreated dyeing?

Dr. GILL: Soap is in general more effective than alkali, but if the cationic groups of the fixative are not quaternary, they will be sensitive to pH, and if the alkali used is sodium hydroxide, the number of cationic groups ionised will in some cases be very small at such high pH values. With such fixatives, therefore, caustic soda will give more bleeding off than soap.

Mr. FOURNESS: Is there any difference in the wet fastness of a dyeing aftertreated with a given agent at different temperatures?

Dr. GILL: Normally the higher the temperature of treatment, the better the fixation, but the difference may vary from quite small to appreciable, depending on the fixative.

Mr. W. ARMFIELD: With the synthetic-resin type of fixing agent, would Dr. Gill expect any mechanical blocking of the dye?

Dr. GILL: We have found no evidence of mechanical blocking with up to 5% fixative. If a resin fixative is used having relatively weak basic groups, the bleeding off of dye at different pH values follows quite closely the pH–titration curve of the resin, which would not be the case if mechanical blocking played much part.

Mr. D. M. WOODS: Will Dr. Gill give an indication of the effect of the dye-fixing agent itself being substantive, say by van der Waals forces? Would not this tend to hold the dye even more

firmly to the fibre, since, as the charges would be partly neutralised, the detergent should have less effect?

Dr. GILL: The logical extension of this is to take the positive sites as being held by valency forces to the cellulose chain, and this case has been discussed in the text to show that virtually no improvement in wet fastness would be obtained as compared with normal fixatives.

Mr. G. ROBINSON: Has Dr. Gill any experience of the difference in effectiveness of the classes of fixation agents discussed when applied to direct dyeings on normal and stretched rayon respectively, i.e. when the proportions of crystalline to amorphous regions differ?

Dr. GILL: Unfortunately we have no experimental information on this point.

Dr. C. M. WHITTAKER: If Class C direct dyes are dyed on viscose rayon in the usual pale lingerie colours to a slightly greater depth than the pattern requires, and are then reduced to the required depth in a boiling soap bath, the resultant dyeings are as fast to washing for lingerie as vat dyeings. Unlike the aftertreatments discussed the dyeings are not depreciated in light fastness.

I have listened with pleasure to the funeral oration on hydrogen bonding.

COMMUNICATIONS

The Relation of the Reflectance of Dyed Fabrics to Dye Concentration and the Instrumental Approach to Colour Matching

E. ATHERTON

The factors governing the reflectance of light by a dyed fabric are discussed, and several theories relating this factor to dye concentration are noted. They may all be reduced approximately to simple reciprocal functions of reflectance. Special reference is made to a theoretical treatment, similar to that given by Kubelka and Munk, for conditions of normal illumination (such as prevail in a spectrophotometer), and the use of this theory in the prediction of matching recipes is illustrated with reference to a simple laboratory instrument.

INTRODUCTION

The way in which the reflectance of a textile is modified by the presence of dye in the fibre is of considerable interest to the dyer, because it is the most important factor determining the amount of dye which must be used to produce a desired shade. Although the practical assessment of dyeings for either dye strength or matching purposes does not require a knowledge of the optical processes involved, such knowledge is desirable when research into the dye–fibre system is contemplated. Thus Preston and Tsien [1], by studying the effect of dye concentration on reflectance, have been able to calculate the orientation of dye molecules from the dichroism of viscose rayon dyeings.

The primary aim of investigating the reflectance of dyed textiles is the development of a mathematical function of reflectance which bears a linear relation to dye concentration, since such a function would be analogous to the optical density (logarithm of the reciprocal of the transmission) of a transparent coloured medium. A function of this type would also be additive for mixtures of non-interacting dyes, and would be of use as a basis for match prediction. Such a function has, in fact, already been used for this purpose by Selling [2] and, although the equation he used is empirical and may be applicable only to a limited number of substrates and dyes, his work is an example of the possible applications of this type of analysis. Many other authors have reported functions which give linear plots against concentration.

Excluding empirical functions from discussion, the most interesting are those of Guelke and Fitzsimons [3], Preston and Tsien [1], Kubelka and Munk [4], and Pineo [5]. The first two treat the fabric as a series of homogeneous coloured laminae characterised by absorption and reflection factors. The reflection of the whole system of laminae is calculated as the sum of a power series and, by the use of Beer's law connecting absorption factor and dye concentration, a linear relation between concentration and a reflectance function is obtained. Although the model adopted is only an approximation to the real structure of the fabric, both theories give a reasonable fit with experimental data; but the Preston and Tsien function is the more accurate, because it takes into account the absorption of back-reflected light in the fabric, a factor which is ignored in the Guelke and Fitzsimons theory.

The equation of Kubelka and Munk [4], which has been widely used in colour assessment [6–8], is not based on a detailed physical model. Their theory assumes that the material is homogeneous and uniformly coloured, that its reflectance is measured under perfectly diffuse illumination, and that the reflected flux also is perfectly diffuse. On these assumptions a simple reflectance function is developed which is equal to the ratio of the absorption coefficient to the scattering coefficient of the fabric. If the dye and the fabric are assumed to absorb independently and the dye does not affect the scattering by the fibre, this ratio

may be divided into two terms and varies linearly with dye concentration. The Pineo theory [5] is based upon the same assumptions, but considerations of the distribution of scattered light lead to a modified form of the Kubelka–Munk reflectance function containing empirical constants the values of which may be adjusted to suit the particular textile studied.

The last two approaches to the problem are probably more satisfactory for general purposes than the lamellar theories, since they focus attention on the absorbing and reflecting properties of the material as a whole without invoking the optical properties of individual fibres. They can apply accurately, however, only if the original assumptions about the optical properties of the fabric are correct. The assumption that the incident light is diffuse is an obvious departure from the normal conditions of measurement, since reflectances are usually measured on a spectrophotometer employing a collimated light beam. Elementary considerations show that this will have an effect on the absorption coefficient as calculated by the Kubelka and Munk equation. The reflection observed is due to normally incident light, which takes the shortest possible path length through the fabric to any particular volume element. The probability of encounter with fibres along such a path to the element is a minimum, and therefore the intensity of light reaching the element is greater than any other value it could have if the same intensity of incident light were distributed in some other way. The theory assumes, however, that the incident light is diffuse, which would correspond to longer paths to the element and hence to increased absorption. On this basis the contribution of the volume element to the diffusely scattered light of the whole fabric is smaller than is actually the case when the incident light is parallel. If the equation is applied without modification, under conditions of parallel illumination, the calculated absorption coefficient is lower than the true value because of the greater path lengths assumed in the equation. The fact that the theory affords linear plots of a reflectance function against concentration indicates that the factors controlling the diffusity of light within the medium are not changed appreciably by additions of dye.

At first sight it is surprising that the diverse treatments which have been used to analyse the reflection of light by dyed textiles have given apparently different functions of reflectance which are linearly related to dye concentration and yet which have all received experimental verification. As a result of an investigation into a model similar to that of Kubelka and Munk but involving collimated incident light, it became clear that all the functions discussed approximate to the form predicted by this modified theory. Because of the similarity of the treatment to that used by Kubelka and Munk, a shortened form of the analysis is given, and emphasis is laid on the comparison of the resulting reflectance function with those previously mentioned.

MODIFIED KUBELKA AND MUNK THEORY ALLOWING FOR COLLIMATED INCIDENT LIGHT

The treatment follows closely that given by Kubelka and Munk, with the difference that the light is assumed to be a parallel beam, incident normally. For a rigid mathematical treatment it is necessary to consider three light fluxes within the medium (Fig. 1)—

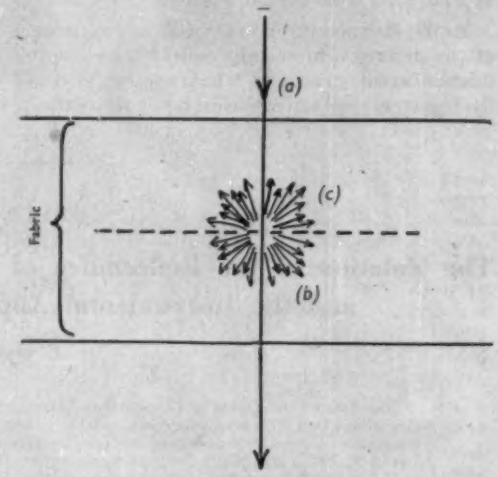


FIG. 1

(*a*) A collimated flux travelling in the direction of incidence

(*b*) A diffusely scattered flux in the same direction

(*c*) A diffusely scattered flux in the opposite direction.

The differential equations applying to such a system involve three terms, and it is therefore not possible to solve them simply as in the Kubelka and Munk theory. By ignoring flux (*b*) we can simplify the equations without invalidating the theory to any great extent, since, for such a flux to emerge finally from the front face of the fabric, at least two encounters of the incident light with fibres would be necessary, as compared with the one encounter which is the minimum requirement for the derivation of flux (*c*). With the approximation that the downward diffuse flux is negligible, the theory for collimated incident light (subsequently called the "C.I." theory) may be developed in a similar way to the Kubelka and Munk theory (subsequently called the "K.M" theory), as follows.

The fabric is represented by a parallel-sided, coloured scattering medium, having a collimated beam of monochromatic light incident normally on one face. The intensity of the collimated beam at a distance x within the medium from the surface of incidence is I_{cx} and the intensity of the oppositely directed diffuse flux at the same point is I_{sx} (Fig. 2).

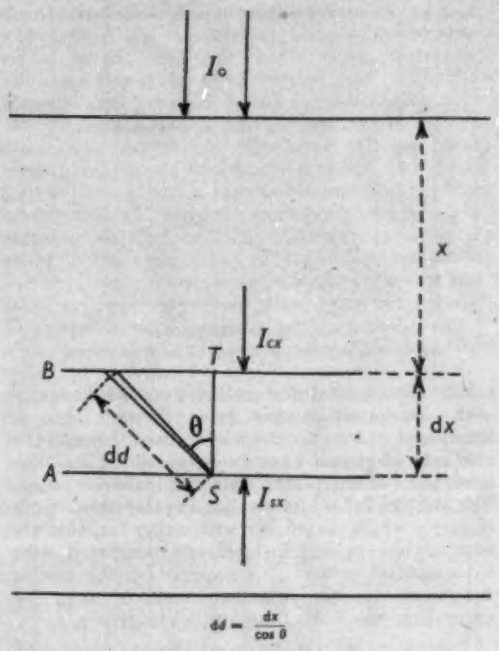


FIG. 2

The reduction of the two fluxes due to passage through an elementary layer of thickness dx at x may be written—

$$-dI_{cx} = I_{cx}(K+S)\,dx \qquad \text{(i)}$$

$$-dI_{sx} = I_{sx}(K+S)\,\overline{dd} - I_{cx}S\,dx \qquad \text{(ii)}$$

where K and S are the absorption and scattering coefficients of the material and $\overline{dd}$ is the mean path length of diffuse light through the layer of thickness dx; $\overline{dd}$ is always greater than dx, since much of the diffuse light travels through the layer along paths greater in length than dx. The evaluation of $\overline{dd}$ is discussed in an Appendix (p. 397). Writing $\overline{dd} = l\,dx$, where l is the diffusity factor, and rearranging (i) and (ii)—

$$-\frac{dI_{cx}}{I_{cx}} = (K+S)\,dx \qquad \text{(iii)}$$

$$-\frac{dI_{sx}}{I_{sx}} = (K+S)\,l\,dx - \frac{I_{cx}}{I_{sx}}\,S\,dx \qquad \text{(iv)}$$

Subtracting (iv) from (iii) and writing $r = I_{sx}/I_{cx}$, the reflectance at depth x inside the medium—

$$d\ln r = \frac{dr}{r} = \left[\frac{1}{r} - \left(\frac{K}{S}+1\right)(l-1)\right] S\,dx$$

or

$$dr = \left[1 - r\left(\frac{K}{S}+1\right)(l-1)\right] S\,dx \qquad \text{(v)}$$

Separating the variables and integrating between R and R' gives—

$$\ln\left[\frac{1 - R'\left(\frac{K}{S}+1\right)(l-1)}{1 - R\left(\frac{K}{S}+1\right)(l-1)}\right] = \left(\frac{K}{S}+1\right)(l-1)SX \qquad \text{(vi)}$$

where R and R' are the reflectances at the front and back surfaces respectively, and X is the thickness.

If an infinitely thick layer of the medium is considered (i.e. $X \to \infty$)—

$$R_\infty = \frac{1}{\left(\frac{K}{S}+1\right)(l-1)}$$

$$\text{or}\quad \frac{K}{S} = \left[\frac{1}{R_\infty(l-1)} - 1\right] \qquad \text{(vii)}$$

where R_∞ is the reflectance of an infinitely thick layer of the medium. Substitution from (vii) in (vi) gives—

$$\ln\left(\frac{1 - R'/R_\infty}{1 - R/R_\infty}\right) = \frac{SX}{R_\infty} \qquad \text{(viii)}$$

If the medium is backed by a surface of zero reflectance $R' = 0$ and R is denoted by R_0. Equation (viii) then becomes—

$$S = \frac{R_\infty}{X}\ln\left(\frac{1}{1 - R_0/R_\infty}\right) \qquad \text{(ix)}$$

The K.M. equations to be compared with (vii) and (ix) are—

$$\frac{K}{S} = \frac{(1 - R_\infty)^2}{2R_\infty} \qquad \text{(x)}$$

and

$$S = \frac{R_\infty}{X(1 - R_\infty^2)}\ln\left[\frac{1 - R_0R_\infty}{1 - R_0/R_\infty}\right] \qquad \text{(xi)}$$

A slight modification of the C.I. equations enables the theory to be applied to conditions of inclined incident light, which have been used by several workers. If light is incident at an angle $\cos^{-1}(1/n)$ to the normal, the intensity incident on the medium is reduced to one-nth and the final equation becomes—

$$\frac{K}{S} = \left[\frac{1}{R_\infty(ln - 1)} - 1\right] \qquad \text{(xii)}$$

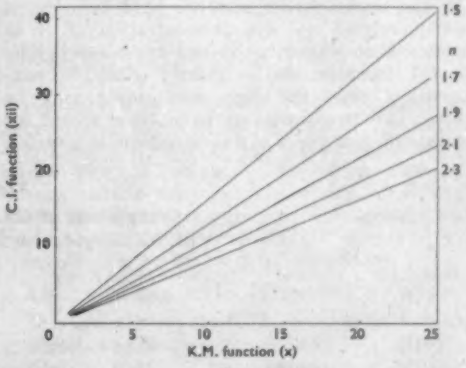


FIG. 3

In order to investigate the effect of changing the angle of illumination, the above function was plotted against the K.M. function (x) by varying R_∞, each plot (Fig. 3) corresponding to a different value of n ($l = 1{\cdot}5$ as shown in the Appendix, p. 397). The gradients of the resulting lines were plotted against n (Fig. 4), and the value of n ($= 1{\cdot}99$) was selected which corresponded to a gradient of 1·0. For this value of n the C.I.

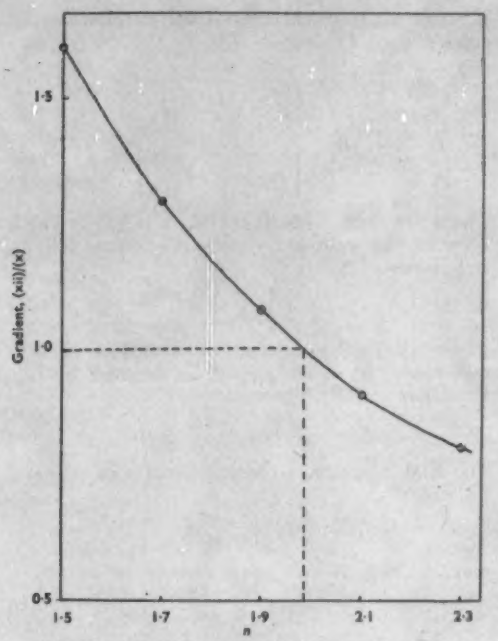


FIG. 4— Gradient of C.I./K.M. Plot as a Function of n

equations for inclined illumination correspond to the K.M. equation, and $n = 1{\cdot}99$ corresponds closely to incidence at 60° to the normal. This value is in agreement with that predicted by Kubelka [9] in a mathematical analysis of turbid media illuminated obliquely.

DISCUSSION OF THE C.I. REFLECTANCE FUNCTION

It was shown earlier that the K.M. function is linearly related to dye concentration if it is assumed that scattering by the dye is negligible. The C.I. function also is linearly related to concentration, since the same assumption may be made. The two functions have been tested for several different types of dye on various substrates,

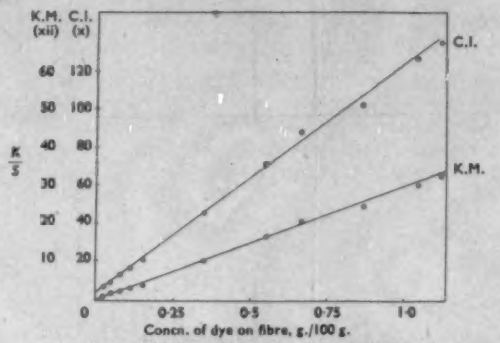


FIG. 5— Naphthalene Fast Orange 2G on Wool Flannel

and no significant difference between their linearity with concentration has been found. This is illustrated in Fig. 5. Regression lines through the two sets of points have been calculated and the deviations of the points from the lines computed. The results (Table I) show that any deviations from linearity which may occur with either function are comparable, or are insignificant compared with experimental errors. The reasons for the similar behaviour of the functions may be seen by expanding the K.M. function (x) to give—

$$\frac{K}{S} = \frac{1}{2\,R_\infty} - 1 + \frac{R_\infty}{2} \qquad \text{(xiii)}$$

which is essentially a reciprocal function of reflectance for all but large values of R_∞. To this extent the C.I. function may be considered as an approximation to the K.M. function.

Several other functions will now be compared with the C.I. function.

THE PRESTON–TSIEN FUNCTION [1]

This function is as follows—

$$\log\left[\frac{\frac{1}{R_\infty}(1-2\rho)+\frac{1}{\rho}-4}{\frac{1}{\rho}-\frac{1}{R_\infty}}\right] = B\,c \qquad \text{(xiv)}$$

where ρ is the coefficient of reflectance for the fibre–air interface and B is a constant involving

TABLE I

Comparison of C.I. and K.M. Functions

(Wool flannel dyed with Naphthalene Fast Orange 2G)

Concn. of Dye (c g./g.)	R_∞	K/S K.M.	K/S C.I.	$\Delta(K/S)$* K.M.	$\Delta(K/S)$* C.I.	$\frac{\Delta(K/S)}{c} \times 10^{-3}$† K.M.	$\frac{\Delta(K/S)}{c} \times 10^{-3}$† C.I.
0·0113	0·0149	32·56	132·2	32·46	129·7	2·88	11·49
0·0105	0·0160	30·26	123·9	30·16	121·4	2·87	11·58
0·0087	0·0197	24·39	101·1	24·29	98·6	2·81	11·32
0·0067	0·0230	20·78	85·9	20·65	83·4	3·08	12·42
0·0056	0·0284	16·62	69·4	16·52	66·9	2·95	11·92
0·0035	0·0448	10·18	43·6	10·08	40·1	2·88	11·47
0·0015	0·103	3·92	18·47	3·82	15·97	2·55	10·65
0·0010	0·126	3·02	14·8	2·92	12·3	2·92	12·30
0·00075	0·165	2·11	11·1	2·01	8·6	2·68	11·47
0·00040	0·245	1·17	7·16	1·07	4·66	2·67	11·65
0·00020	0·336	0·66	4·95	0·56	2·45	2·80	12·25

* $\Delta(K/S)$ represents the difference between the calculated values of K/S (columns 3 and 4) and the value of K/S for the undyed substrate (= 0·1 (K.M.) and 2·5 (C.I.)).

† Standard errors in columns 7 and 8 are 5·23 and 4·06% respectively.

the absorption coefficient of the dye. The function was derived for the interpretation of reflectance measurements on dyed viscose rayon filaments. The measurements were carried out with the filament axes oriented in the same direction, and gave information on the extent of molecular orientation within the filaments. Under these conditions, the use of a lamellar model as a basis for the theory was a reasonable one, but for the interpretation of measurements on fabrics it is not quite so satisfactory. The difficulty in using it arises in the assignment of a value for ρ which will apply to a fabric, since it is the surface of the fabric and not that of the fibre which is relevant. Thus wool flannel composed of fibres each having ρ = approx. 0·04 will have a much smaller surface reflectance owing to the disorientation of the individual fibres. The difficulty is to be expected, however, since the more detailed the model used in terms of fundamental quantities (such as ρ), the more difficult the application to a real fabric. This implies no criticism of the equation, which was not, in the first instance, intended to be applied to fabrics.

There is no easy method of determining a value of ρ for a fabric, since it is of the order of 0·01 or less. It is apparent, however, that for a value of ρ less than 0·01, the denominator in the Preston–Tsien function will be substantially constant for all but very small values of R_∞. If $1/R_\infty$ is neglected in the denominator, equation (xiv) may be written—

$$\frac{\frac{1}{R_\infty}(1-2\rho)+\frac{1}{\rho}-4}{\frac{1}{\rho}} = e^{Bc} \qquad \text{(xv)}$$

e^{Bc} may also be approximated to $(1 + Bc)$ for all but large values of c. The errors involved in making the two approximations will in fact tend to cancel, and equation (xv) may be written—

$$\frac{1}{R_\infty}\rho(1-2\rho)-4\rho = Bc \qquad \text{(xvi)}$$

which is essentially a relation between the reciprocal of the reflectance and the concentration (cf. equation (vii)).

The importance of the value chosen for ρ in the Preston–Tsien reflectance function is shown in Fig. 6, in which this function is plotted against dye concentration for three different values of ρ, using the data for Naphthalene Fast Orange 2G. It can be seen that for ρ = 0·04 (the approximate reflection coefficient of individual wool fibres) the plot has marked curvature, which is less, though still evident, for ρ = 0·01. For ρ = 0·005 a straight line is obtained, indicating that the surface reflectance of a wool fabric may be as low as 0·5%.

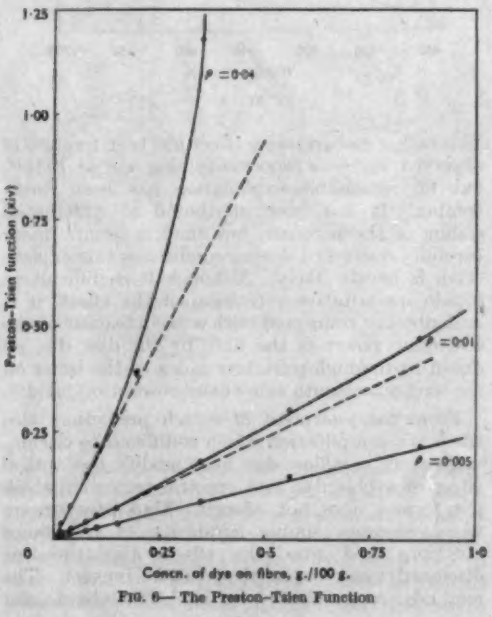


FIG. 6— The Preston–Tsien Function

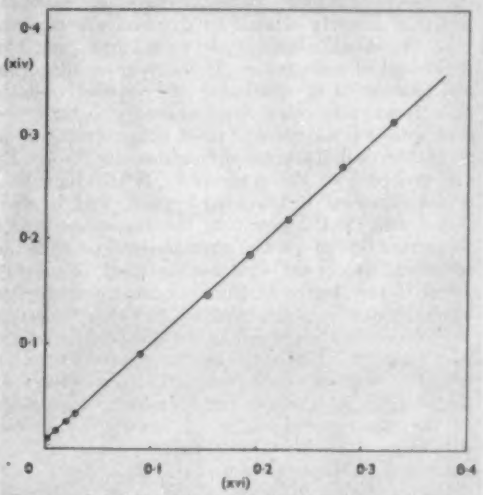


FIG. 7— Plot of Complete against Approximate Preston–Tsien Reflectance Function (ρ = 0·005)

A comparison is made between the complete and approximate forms of the reflectance function in Fig. 7, using a value of ρ = 0·005. Again a good linear plot is obtained, indicating that the approximations made in transforming the Preston–Tsien function to the reciprocal reflectance form do not appreciably modify its linearity with concentration under practical conditions.

THE PINEO FUNCTION [5]

This is similar to the K.M. function but contains two empirical factors—

$$\frac{K}{S} = \frac{[1-(R_\infty - p)^{1/n}]^2}{2(R_\infty - p)^{1/n}} \qquad \text{(xvii)}$$

where the values of p and n are chosen to give the best fit with experimental results. Values of n do not differ appreciably from unity for most fabrics, so that it may to a first approximation be disregarded. Thus the function is identical with the K.M. (x) except for p, which is included to allow for surface reflection by the fabric. Although this represents an improvement, particularly for high dye concentrations with correspondingly low

reflectances, for most purposes it is not important. The Pineo function may therefore be regarded as an empirically modified version of the K.M., which has already been shown to correspond closely to a reciprocal reflectance form.

THE SELLING FUNCTION [2]

The following empirical equation was proposed by Selling [2] for the colour analysis of dyed wool—

$$a\,c = \left[\frac{R_w}{R_\infty} - 1{\cdot}1\right]^{1{\cdot}1} \qquad \text{(xviii)}$$

in which a is a constant and R_w is the reflectance of the undyed fabric. The similarity to equation (vii) is evident, apart from the exponent 1·1, the significance of which is not readily apparent.

COMPARISON OF VARIOUS FUNCTIONS

It can be seen from the foregoing discussion that the essential feature of any reflectance function which is linearly related to dye concentration is that it should include a term involving the reciprocal of reflectance. If the various functions are considered as consisting of reciprocal reflectance terms plus other supplementary factors, one may question the value of these latter terms, which increase computational difficulties, in improving the precision of the equations. If the Kubelka–Munk equation is taken as typical, and is compared with the C.I. equation, the deviations from a regression line of a set of experimental points is no different whichever equation is used (Table I). Even if the degree of linearity provided by one function were slightly superior to that obtainable with the others, possibly at the expense of increased computational difficulty, the uncertainties which arise in practice, owing to anomalous effects at wavelengths away from the absorption maximum of the dye, would make the extra, hard won precision of little value. These effects will be discussed in the next section.

APPLICATION TO COLOUR MATCHING

Apart from the aesthetic satisfaction of deriving a reflectance function whose dependence on dye concentration is strictly linear, no outstanding advantages are obtained, because the uses which are made of such a function are based upon the dyeing process, which is not itself strictly reproducible. Thus it is of little value to predict to a small fraction of 1% the concentration of a dye on a fabric which would be required to produce a given colour if, for example, the exhaustion of the dyebath cannot be controlled to better than 1%.

The most general uses which have been made of reflectance functions are the estimation of strength and the prediction of matching recipes. In measuring the strength of a dye, a calibration curve derived from measurements on the standard dye is used. These measurements are plotted (in the form of one of the previously mentioned reflectance functions) against concentration, giving a linear plot. The strength of a batch of the same dye may then be calculated by a single-strength dyeing. There is no advantage in using a reflectance function for this purpose, since a simple plot of reflectance against concentration would serve equally well.

The second use is more important, and satisfactory methods of predicting matching recipes have been devised [2, 10, 11]. From the results published, however, it is clear that discrepancies arise, the causes of which are not fully discussed. The degree of success so far achieved does not indicate that the methods can replace a trained colourist, and it is the present author's opinion that no method based on reflectance functions of the type discussed can achieve the requisite precision. This is because anomalous effects occur in practice, which cannot be allowed for. Thus, although the expected behaviour is found in the wavelength range corresponding to the maximum absorption of a dye, the reflectance curves in other spectral regions are irrational [12–14]. In the example shown (Fig. 8) the most heavily dyed pattern has the highest reflectance at wavelength greater than 540 mμ., while the undyed pattern has the lowest.

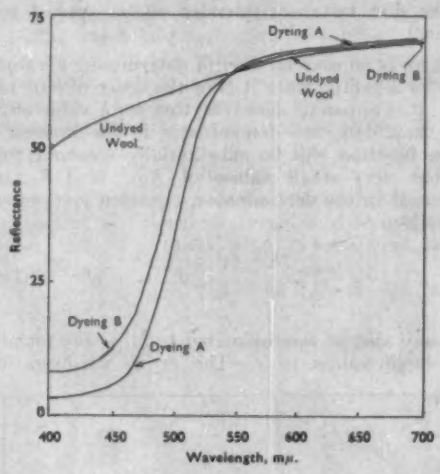


FIG. 8

This rather embarrassing effect has been frequently observed, and was reported as long ago as 1934 [13], but no reasonable explanation has been forthcoming. It has been attributed to differential soiling of the patterns, but since it occurs under carefully controlled dyeing conditions, this explanation is hardly likely. Although it is difficult to obtain quantitative repetition of the effect, it is undoubtedly connected with a modification of the scattering power of the fibre by the dye, due no doubt to the high refractive index of the latter on the long-wavelength side of the absorption band [15].

From the viewpoint of match prediction, this effect is a complication which is difficult to circumvent, since a yellow dye may modify the optical effect of a blue dye in a spectral region in which the former does not absorb. Thus any system which assumes simple additivity of reflectance functions and overlooks effects like the one discussed must be to that extent inexact. The methods reported by Selling [2], Pritchard and

Stearns [11], and Derby [10] and the one to be described in the present paper are therefore limited in their accuracy, and must be considered as rapid methods of obtaining rough matching recipes.

The only differences between the various systems are the methods of manipulating the data available on the component dyes to be used in making the match. Stearns [11] and Derby [10] utilise the "R" cam provided as an accessory to the General Electric recording spectrophotometer, which causes the instrument to plot—

$$\log \frac{(1 - R_\infty)^2}{2R_\infty}$$

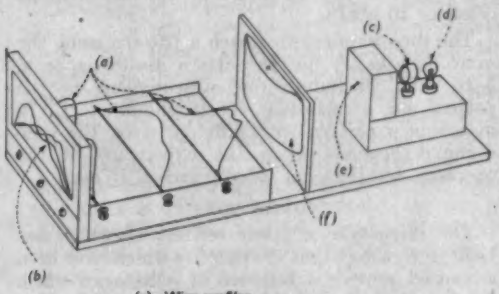
against wavelength. The curves representing the dyes to be used in the matching, at known concentrations on the fibre, are added graphically by means of a logarithmic scale and compared with the curve representing the dyeing to be matched.

The principle of this process is illustrated by an instrument constructed to provide a rapid method of matching wool dyeings using Lissamine Fast Yellow 2G, Azo Geranine 2G, and Solway Blue BN. These dyes were selected because they cover a wide range of colours and are compatible when dyed in mixtures on wool.

Dyeings on wool flannel were prepared of each dye at several known concentrations, and reflectance curves were measured with the recording spectrophotometer. The undyed substrate also was measured, and curves showing the variation of the function—

$$\left[\left(\frac{1}{R_\infty}\right)_{\text{Dyeing}} - \left(\frac{1}{R_\infty}\right)_{\text{Substrate}}\right] \qquad \text{(xix)}$$

with wavelength were prepared. Lengths of 36-gauge wire were bent to the shape of the curves and mounted on three parallel axles as in Fig. 9.



(a) Wire profiles
(b) Shadows of profiles
(c) Condenser
(d) Car bulb
(e) Pinhole
(f) Converging lens

FIG. 9

Light from a 36-watt car bulb was rendered parallel by means of a condenser, a pinhole, and a large-aperture collimator lens, so as to cast shadows of the three wires on a screen. A scale of the above function (xix) was placed over the screen, so that ordinates of the curves could be added conveniently. The axles on which the curves were mounted could be rotated so that the shadows could be uniformly expanded or contracted to simulate the effect of varying dye concentration.

A matching recipe for a given pattern is predicted by drawing a curve of the reflectance function, corrected for substrate, against wavelength on the scale. The wires are adjusted progressively, additions being carried out at several ordinates between settings, until positions are obtained for which the addition curve gives a close fit to the curve to be matched. The concentrations of the three dyes are then read from the angular positions of the axles. The instrument is shown in Fig. 10.

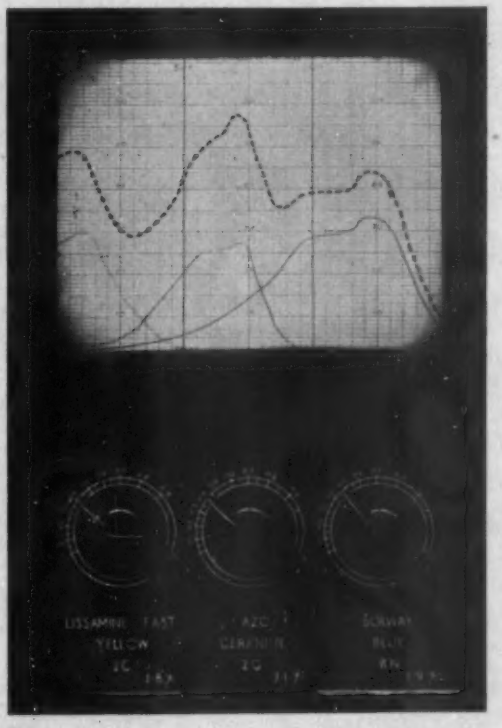


FIG. 10

No great difficulty has been experienced in matching patterns dyed with mixtures of the same three dyes. The results of several measurements are given in Table II.

All the matching recipes were dyed and the resulting patterns compared with the originals. The patterns were submitted to a number of experienced colourists, and the agreement was judged to be better than the first speculative matching which would be obtained by normal laboratory methods. The patterns were also compared using the recording spectrophotometer, by calculating the number of just perceptible colour steps between the original and the match. The average value was approximately two steps.

TABLE II

Predicted Matching Recipes

Pattern No.	Dye Concentration (% on wt. of fibre)					
	Solway Blue BN		Azo Geranine 2G		Lissamine Fast Yellow 2G	
	Actual	Calc.	Actual	Calc.	Actual	Calc.
1	0·2	0·216	0·5	0·500	0·5	0·536
2	0·4	0·400	0·5	0·510	0·5	0·500
3	0·6	0·600	0·5	0·500	0·5	0·520
4	0·8	0·780	0·5	0·536	0·5	0·530
5	1·0	0·912	0·5	0·532	0·5	0·528
6	0·5	0·496	0·2	0·204	0·5	0·528
7	0·5	0·516	0·4	0·410	0·5	0·560
8	0·5	0·524	0·6	0·570	0·5	0·528
9	0·5	0·516	0·8	0·804	0·5	0·541
10	0·5	0·530	1·0	1·000	0·5	0·530
11	0·5	0·530	0·5	0·528	0·2	0·216
12	0·5	0·516	0·5	0·532	0·4	0·403
13	0·5	0·524	0·5	0·526	0·6	0·580
14	0·5	0·523	0·5	0·529	0·8	0·764
15	0·5	0·518	0·5	0·525	1·0	0·840
16	0·33	0·340	0·5	0·532	0·75	0·770
17	0·5	0·520	0·75	0·744	0·75	0·810
18	0·75	0·718	0·75	0·726	0·5	0·572
19	0·1	0·126	0·1	0·096	0·2	0·202
20	0·1	0·124	0·1	0·098	0·3	0·296
21	0·2	0·226	0·2	0·197	0·4	0·482
22	0·2	0·226	0·2	0·196	0·6	0·572
23	0·2	0·232	0·6	0·562	0·2	0·168
24	0·2	0·216	0·4	0·368	0·2	0·172
25	0·2	0·226	0·1	0·100	0·1	0·070
26	0·3	0·314	0·1	0·102	0·1	0·114
27	0·4	0·396	0·2	0·182	0·2	0·180
28	0·6	0·588	0·2	0·210	0·2	0·194
29	0·1	0·126	0·1	0 100	0·1	0·0996
30	0·2	0·234	0·2	0·199	0·2	0·199
31	0·1	0·122	0·3	0·286	0·1	0·084
32	0·1	0·128	0·2	0·202	0·1	0·126

When the dyeing to be matched contains dyes different from those to be used in matching, the problem is more complicated. The difficulty arises in deciding when the addition curve has been adjusted to give the best fit with a given curve, since it is impossible with the present instrument to make allowance for the illuminant or the varying sensitivity of the eye. When matching a curve, if deviations are unavoidable, it is best to arrange that in spectral regions where the reflectance function is large (low reflectance) the curve representing the match shall rise to higher values than the curve of the pattern, if by so doing the parts of the curves at medium and low values of the reflectance functions can be made to agree. This is because these spectral regions correspond to the maximum reflectance of light by the pattern, and are most important in determining the colour. Another factor in improving the accuracy of the method is the agreement between the curves at wavelengths 450, 550, 610 mμ., where the weighted tristimulus functions are maximal. These criteria are particularly useful in matching neutral colours, and if an attempt on these lines fails to produce a reasonable match, it may be concluded that, even if such a match is possible, it is likely to be strongly dependent on the type of illuminant. In this case another combination of dyes should be sought (consistent with the other requirements, such as fastness). This in itself is useful information which might take a lengthy period for all but a skilled colourist to obtain.

The time required to match a pattern using the same dyes as are on the pattern itself is approx. 5–10 min. (including the measurement of the pattern on a recording spectrophotometer). In matching a pattern with different dyes, the time required depends upon the experience of the operator, but does not usually exceed 15 min.

CONCLUSIONS

The discussion of fabric reflectance which has been given shows that the theories which have been advanced provide a function of reflectance which for practical purposes is linearly related to dye concentration. The Kubelka–Munk function has been considered in some detail, and a modification of the theory has been made in order to bring it more into line with conditions of measurement which occur in the G.E. recording spectrophotometer.

The resulting reflectance function, which is simpler than the original, has been shown to be equivalent to it for purposes of match prediction. The modified function, moreover, appears to be the most important part of the other reflectance functions which have been discussed, since they all approximate to it. Unless new applications of

reflectance functions, requiring high precision, arise in future, any search for a more elaborate general theory would be unrewarding.

An account has been given of a simple method for predicting matching recipes, and some of the limitations of this type of approach have been mentioned. It appears that these methods form a basis for obtaining approximate matching recipes more quickly and conveniently than by ordinary laboratory methods. The wider application of such systems of analysing colour problems is likely to await the appearance of a recording spectrophotometer for reflection measurements, at a reasonable price, on the British market. It may be that some of the existing multifilter colorimeters would be satisfactory for this purpose, in view of the inaccuracies already inherent in the method, but this point has not been checked.

It is extremely unlikely, however, that the methods discussed will ever replace the colourist, since the last small additions of colour which must be made to improve a dyeing, to which the eye is sensitive— which in fact are necessary only because of this sensitivity— will remain the field of the skilled colourist. It is apparent, however, that the methods described can already provide him with a useful tool, which will ease his burden for those aspects of his work which do not require the full analytical power of the trained eye.

Appendix

CALCULATION OF THE MEAN OPTICAL PATH LENGTH FOR DIFFUSE LIGHT PASSING THROUGH AN ABSORBING LAYER

The calculation has been carried out without including the effect of scattering in the layer, since on average there will be no increase of path length due to scattering, as this is a random process.

In order to calculate the mean optical path length it is necessary to weight each particular path by an amount depending on the appropriate property of the medium. The implication of this may be seen later when the system has been defined. Let S (Fig. 2) be a point source which emits light energy equally in all directions between $\theta = 0$ and $\theta = \pi/2$. Initially the problem is to calculate the total light energy emerging from the surface B, but unfortunately the simple differential equation used in deriving Beer's law for solutions is not applicable; i.e. it is incorrect to write—

$$-\mathrm{d}I_\theta = I_\theta K \,\mathrm{d}d\,\mathrm{d}\theta \qquad \text{(xx)}$$

where $I\theta\mathrm{d}\theta$ is the energy flux per unit area, per unit time, between angles θ and $\theta + \mathrm{d}\theta$, K the absorption coefficient, and $\mathrm{d}d$ the path length. This is because, as θ approaches $\pi/2$, $\mathrm{d}d$ becomes indeterminate, and equation (xx), which applies only to infinitesimal path lengths, is invalid. The derivation is also complicated because the amount of energy radiated per second at an angle θ to ST is not independent of θ.

The problem may be defined more specifically as follows by considering a hemisphere of radius r and centre S (Fig. 11). If the energy radiated per second in all directions between θ and $\theta + \mathrm{d}\theta$ is $E_\theta\mathrm{d}\theta$ and the intensity within this angular range is I, which is constant, then—

$$E_\theta\mathrm{d}\theta = 2\pi r^2 I \sin\theta\,\mathrm{d}\theta \qquad \text{(xxi)}$$

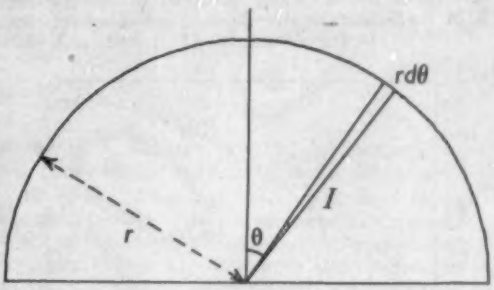


FIG. 11

If the total energy radiated is E, then—

$$E = \int_0^{\pi/2} 2\pi r^2 I \sin\theta\,\mathrm{d}\theta \qquad \text{(xxii)}$$

Integrating and substituting in (xxi) gives—

$$E_\theta = E\sin\theta \qquad \text{(xxiii)}$$

For a path length $\mathrm{d}d$ through the absorbing layer (as shown in Fig. 2)—

$${}_aE_\theta = E_\theta\left[1 - \exp(-K\,\mathrm{d}x/\cos\theta)\right] \qquad \text{(xxiv)}$$

where ${}_aE_\theta$ is the energy absorbed in unit time for emergent light at an angle θ to the normal. Substituting in (xxiv) for E_θ from equation (xxiii)—

$${}_aE_\theta = E\sin\theta\left[1 - \exp(-K\mathrm{d}x/\cos\theta)\right] \qquad \text{(xxv)}$$

$$\text{or } {}_aE = \int_0^{\pi/2} E\sin\theta\left[1 - \exp(-K\mathrm{d}x/\cos\theta)\right]\mathrm{d}\theta \qquad \text{(xxvi)}$$

where ${}_aE$ is the total energy absorbed per unit time.

It is now possible to write an equation—

$${}_aE = E\left[1 - \exp(-K\overline{\mathrm{d}d})\right] \qquad \text{(xxvii)}$$

where $\overline{\mathrm{d}d}$ is the mean path length and is defined by this equation. If equations (xxvi) and (xxvii) are combined, the resulting equation, in effect, determines the weighting which is assigned to each particular path in calculating $\overline{\mathrm{d}d}$. The paths have therefore been weighted according to their absorbing power. The combined equation is—

$$E\left[1 - \exp(-K\,\overline{\mathrm{d}d})\right] = \int_0^{\pi/2} E\sin\theta\left[1 - \exp(-K\mathrm{d}x/\cos\theta)\right]\mathrm{d}\theta \qquad \text{(xxviii)}$$

which, after rationalising and substituting $l\,\mathrm{d}x$ for $\overline{\mathrm{d}d}$, gives—

$$l = -\frac{1}{K\mathrm{d}x}\ln\left\{1 - \int_0^{\pi/2}\sin\theta\left[1 - \exp(-K\mathrm{d}x/\cos\theta)\right]\mathrm{d}\theta\right\} \qquad \text{(xxix)}$$

This integral cannot be evaluated by normal methods, so there is no exact value of the diffusivity factor l. It should also be noted that l has no

absolute significance, since it depends upon K, the absorption coefficient of the medium. This means that for a dyed textile the mean path length is a function of dye concentration, a fact which underlines the difficulty involved in relating the K.M. coefficients to any absolute optical quantities.

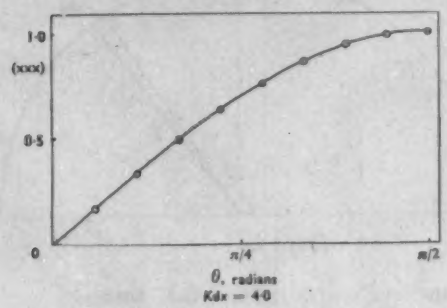


FIG. 12

It is of interest, however, to study the dependence of l upon dye concentration (which effectively determines the value of K for a sheet of given thickness), and this has been done in order to evaluate the C.I. equations. Kdx was given a series of values between 0·1 and 4·0, and the function—

$$\sin\theta\left[1-\exp\left(-Kdx/\cos\theta\right)\right] \qquad \text{(xxx)}$$

plotted against θ from 0 to $\pi/2$, as shown in Fig. 12 for $Kdx = 4{\cdot}0$. The curves were integrated graphically by means of a planimeter, the final area under each curve used in calculations being the average of five measurements (which agreed in general within $\pm$ 0·5%). In each case l was calculated, and the results were plotted against Kdx (Fig. 13). It can be seen that, when Kdx is above 1·0, the value of l is substantially independent of Kdx. Since most of the patterns studied fall in this range of Kdx values, one value of l will represent the optical behaviour fairly accurately, although for lower values the increase in l might be expected to cause deviations from linearity. The

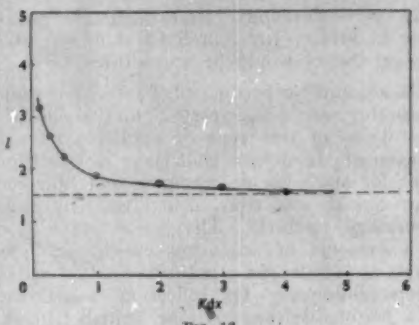


FIG. 13

experimental plots show that these deviations are within the limits of experimental error, since no significant curvature is apparent. The value of l adopted was 1·5 (Fig. 13).

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George William Oldham
Silk Dyer
Moll Spring Dyeworks, Netherton, near Huddersfield

A. THOMSON

On 15th March 1955, at a meeting of the Huddersfield Section of the Society, George Thomson Oldham, the ten-year-old great-grandson of the founder of the firm of George William Oldham, handed to the President of the Society for safe keeping the following books and papers relating to the firm, which had belonged in succession to his great-grandfather, grandfather, and father—

- Book of recipes belonging to George William Oldham and dated 23rd April 1851
- Recipes and patterns of cotton braids 1854
- Recipes, patterns, and costings 1884–1895
- Recipes, patterns, and costings 1895–1905
- Recipes, patterns, and costings 1891–1913
- Recipes and patterns for dyeing spun silk (1899)
- Book of contracts for dyes, chemicals, coal, etc. (1898–1946)
  [extracts appear in the Appendix, p. 401]
- G. W. Oldham, shade card (Patterns 1–300)
- Directions for dyeing the various shades contained in shade card (compiled June 1900)
- Box of separate sheets of recipes, patterns, and costings divided into nineteen different groups as follows—
  - Azoic dyeings
  - Bleach, dye white, and size
  - Catechu browns
  - Diazotised and developed shades (reds and blacks)
  - Dyeings from a soap lather with Glauber's salt
  - Dyeings made in a soap lather containing Glauber's salt with direct and acid dyes
  - Dyeings made from a suds liquor
  - Dyeings in suds or a water with acid
  - Fast-to-boiling shades
  - Logwood blacks on silk
  - Mordant dyeings (on aluminium and chromium)
  - Multicolour, Ombre, and Colour and White (fast-to-boiling shades)
  - "O" dyes
  - Dyeings on rabbit wool
  - Dyeings on silk and wool
  - Sulphur blacks on silk
  - Vat dyes on silk (2)
  - Viscose rayon dyed with (*a*) direct dyes, (*b*) "O" dyes, (*c*) vat dyes
- Miscellaneous notes recording works names and commercial names of dyes dated 1921 and 1926
- Record of fast-to-boiling range dated 1919 and 1922
- Data in connection with bonus scheme, basic rates of pay, etc.
- Book— *Elements of the Art of Dyeing and Bleaching*, by Berthollet and Ure (1841)
- Recipes, instructions, and directions for using N. Spencer Thomas's concentrated dye stuffs (1880), "for dyeing all Colors on all Fibres and on all Fabrics".

An agreement to cover this transfer was then signed by the President (Mr. Fred Smith) on behalf of the Society and by Mrs. M. C. Oldham, Master G. T. Oldham, and Mr. A. Thomson, who had been chemist with the firm for fifteen years.

After the President had presented George Thomson Oldham with a cricket bat as a memento of the occasion, Mr. Thomson, who had recently examined the books and recipes and "translated" the works names of dyes and chemicals into normal commercial names, gave the following brief outline of the history of the firm—

The founder of the firm was George William Oldham, born in Derby in 1830. The family moved to Macclesfield shortly after his birth and jointly earned a living by dyeing silk, so that the boy was familiar with silk and silk dyeing at an early age. When ten years old he started work at 1s. 6d. a week. Trade was, however, very bad, and between the ages of 14 and 25 he worked in no less than 25 places. When 17 he was working at Gaythorne, Manchester, and survived an epidemic of cholera which broke out at that time. Mr. Salkeld, a friend of his employer, opened a dyehouse at Dalton, Huddersfield, and asked for two men. In this way G. W. Oldham came to the Huddersfield district just before the time of the Holmfirth flood, which he well remembered.

His unhappy experience in seeking employment had convinced him that the only way to avoid being out of work was to be in business for himself, and so in 1857 he and his brother Henry opened a dyehouse with a capital of £25 at Spring Vale, near Armitage Bridge. Things went badly at first, and by the end of the first three months their capital had disappeared and they were so poor that G. W. Oldham was compelled to walk to Brighouse carrying a bundle of dyed silk on his back, returning with a fresh lot to dye, a total distance of some thirteen miles. Brighouse and Rastrick at that time were the centre of the waste silk spinning industry. He used to describe how he carried his money in a small bag on a string, and if he met anyone in a lonely place he would stop to rest his silk on a wall and drop the money over, holding the string, so that, if he were attacked, the money could not be found on him. When any possible danger was past, he pulled up the money by means of the string and resumed his journey.

When the Meltham railway was constructed he and his brother were afraid that the vital water supply to their dyehouse might be lost, so they removed to Moll Spring Dyeworks in 1862.

The site of this works was part of the common lands of the "Mag Lordship" of the Manor of Honley and had been awarded as compensation for tithes to the Governors of Clitheroe School

(Lancashire), who owned the profits of the Almondbury Rectory by deed of gift from Philip and Mary in confirmation of a scheme projected by Edward VI.

Early in the nineteenth century a three-storey woollen mill and a cottage stood near the spring in the wood on this land, and during a period when the mill was vacant an old woman named Molly or Moll lived in the cottage, so that the water supply became known as *Moll's Spring*. In 1850 Benjamin Ledger Shaw built a dyehouse on the site of the cottage garden to ensure a supply of very good water, and it was this property that was sold to Henry and George Oldham in 1862.

Ten years later the brothers dissolved partnership, and shortly afterwards G. W. Oldham developed the dyeing of silk in warp form. The warps ran horizontally in the liquor instead of vertically, thus allowing the individual threads to open out and ensuring perfect penetration. He could deal with warps up to 1¼ miles in length, thus greatly economising in time and labour.

G. W. Oldham was prepared to spend both time and money in experimentation. He was the first silk dyer to install electric light and fixed up two 4,000-candlepower arclamps for matching purposes. During one severe winter he erected these lights over the frozen mill dams so that he could skate at night, and crowds of people came to see him cut his monogram on the ice, and also to stare at the wonderful new lighting arrangement, something not previously seen in the district.

The works prospered and at one time he was dyeing enough silk to make 170,000 yd. of silk fabric a week.

G. W. Oldham was very public-spirited, being Honorary Surveyor for Netherton, Village Constable for Honley, a member of the Huddersfield Board of Guardians for 25 years, and a member of the West Riding County Council for 22 years. In 1905 he was made a Justice of the Peace.

The turn of the century marked the development of the "O" dyeing process—a process which was never patented, and was never discovered or used, as far as is known, by anyone else. The normal process for dyeing with basic dyes was to dye the silk from a soap lather or a "broken" (i.e. acidified) suds liquor containing gum removed from silk. The dyeing was then rinsed, aftertreated with tannic acid, and "fixed" with tartar emetic or other antimony salt. By the new process the dye and the tannic acid could be applied together from a fairly strong sulphuric acid liquor. The bath was prepared with 10% sulphuric acid (D.O.V.) and 10% tannic acid (B.P. quality) on the weight of the silk, and dyeing proceeded normally, additions being made to bring to shade as required. The lake normally formed was held in solution, which enabled perfect penetration and evenness to be achieved as well as fastness to rubbing, because the dye–tannin lake was formed *in situ*. Because the lake was already formed, matching was also very much easier. The dyeing was then rinsed, "developed" with antimony salts, rinsed again, and soaped at the boil in the usual way. This process was a great advance on any method previously possible, and the results were faster to both light and washing.

Records exist of the use of this process early in 1901 but, unfortunately, notes on the experimental work which led to this discovery appear to have been lost. Trials were nearly abandoned at one time because the German chemist (Bindschedler), who was asked to carry out the experimental work, happened to choose for his trials Methylene Blue 2B, which is the only dye with which the process will *not* work. This dye contains a metal, and it is probable that the zinc salt interfered with the reaction.

The success of the "O" dyeing process contributed to the changeover from the plush and carpet trades to the "milling" trade, i.e. to the production of silk to be used for pin-stripes in wool suitings, etc. Moreover, the dyehouse was in an excellent geographical position, as it was within easy reach of Huddersfield, Brighouse, Bradford, and the other main centres of the wool trade in the West Riding.

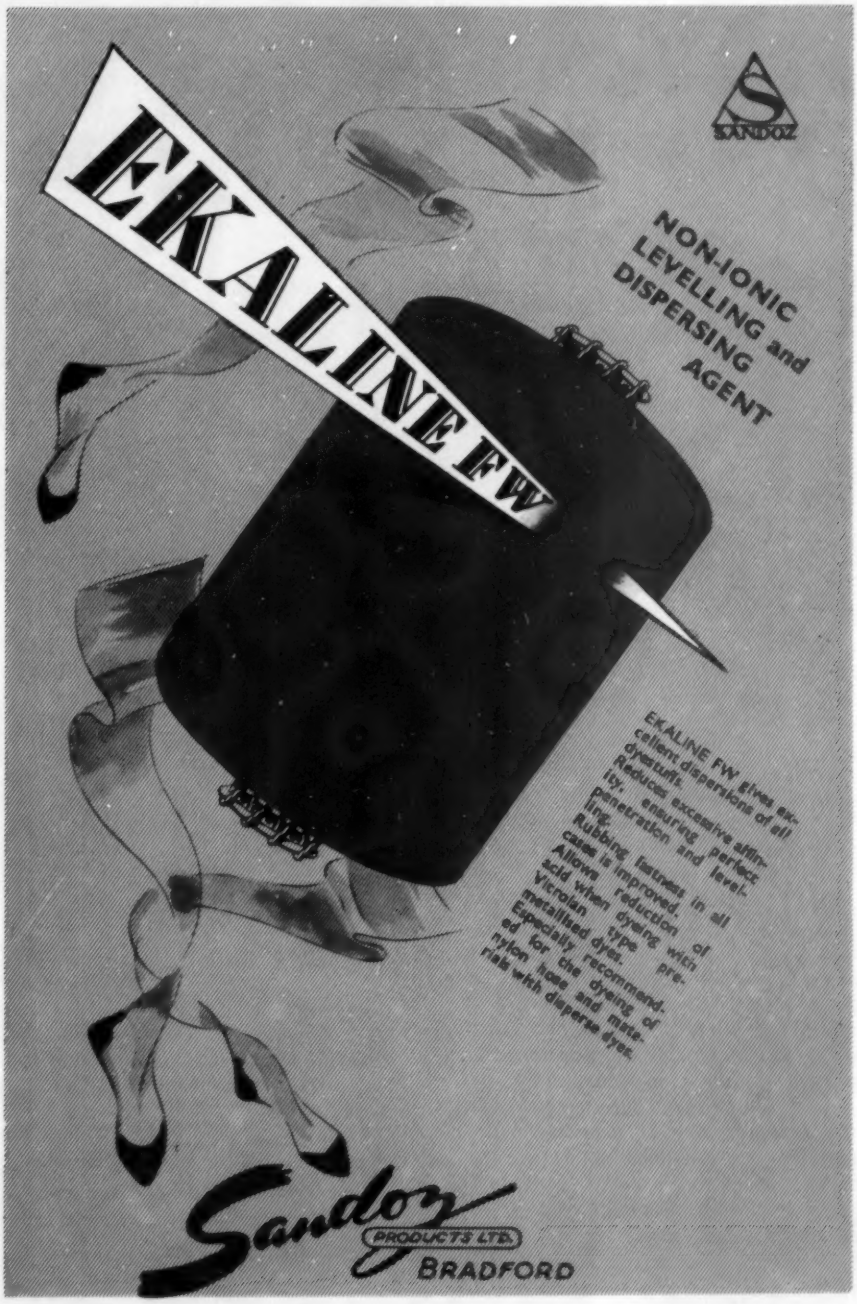
All three generations of the Oldham family were alike in their eagerness to try out new types of dyes as they became available. Records exist of early experiments with the ice dyes (ingrain dyeings), and by 1912 silk was being dyed in bulk with indigoid vat dyes. Anthraquinone vat dyes were in general use some ten or eleven years later, by which time it was realised that silk was far more resistant to caustic soda than had been originally supposed. During the First World War G. T. Oldham bought a 10-lb. tin of Helindon Orange R powder for £107. This was specially stored in the laboratory, much against the wishes of the cashier, who was of the opinion that it ought to be in the safe. Successive lots of silk were dyed in the same bath to ensure complete exhaustion of the dyebath.

The introduction of the solubilised vat dyes presented a further challenge, and a method of development just below the boil was worked out using sodium nitrite and sulphuric acid for a period of 25 sec. only. As the whole operation was done by hand at that time, it will be appreciated that a stop-watch and much preparation were necessary.

Sulphur dyes were originally dyed by the Lodge–Evans process, but a method was worked out using hydrosulphite and ammonia which enabled colours from pale greys to blacks to be dyed in the dyehouse in copper vessels.

Perhaps the work involved when the suitability of the azoic dyes for silk was investigated will further illustrate the thoroughness which the firm adopted when approaching a new problem. The question of the greater suitability of bases or salts was first investigated; then dyeings of over 750 combinations of coupling and diazo components were made and tested for fastness to light, degumming, and peroxide bleaching, before a final selection of a dozen or so suitable combinations was made.

G. W. Oldham died in 1914 and was succeeded by George Thompson Oldham as head of the firm.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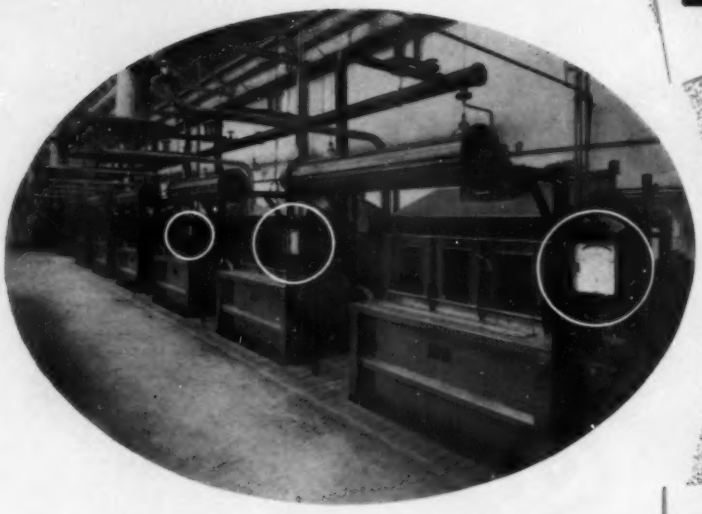

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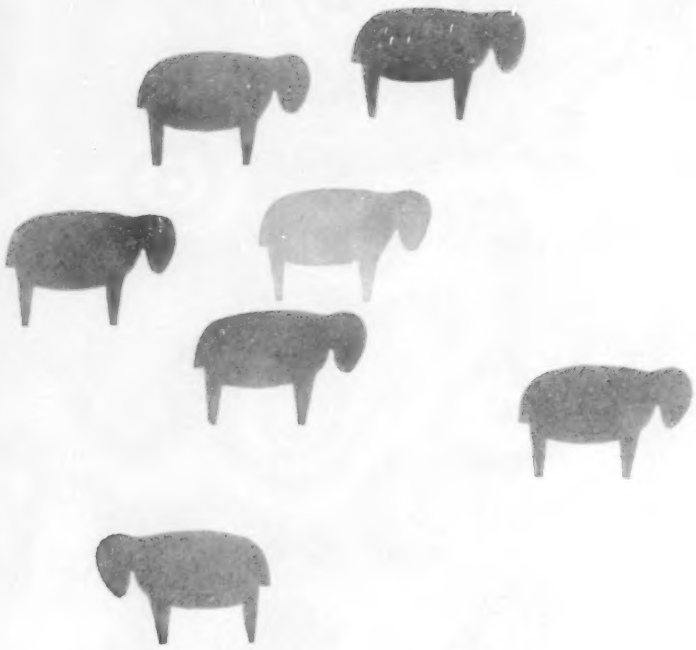

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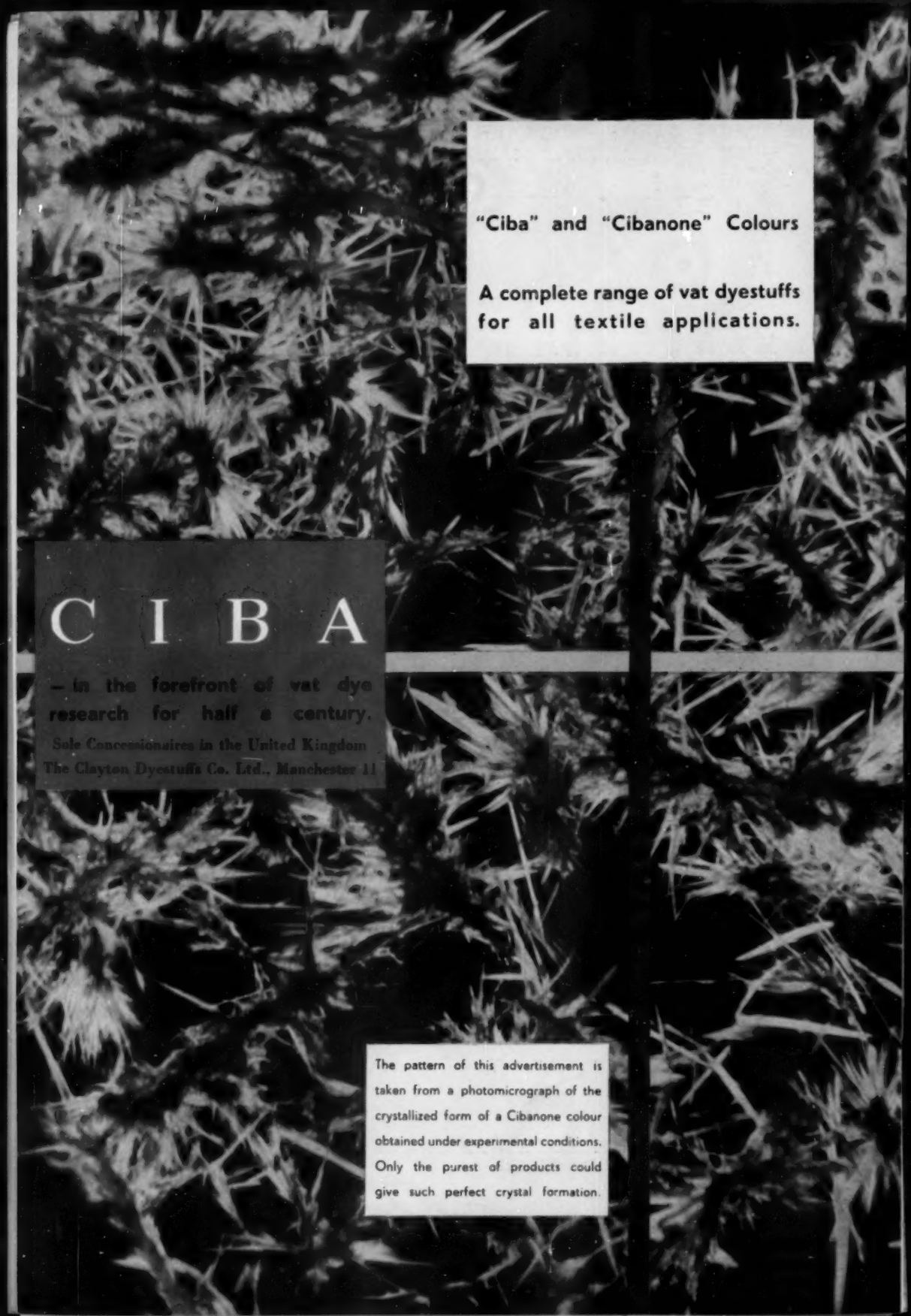
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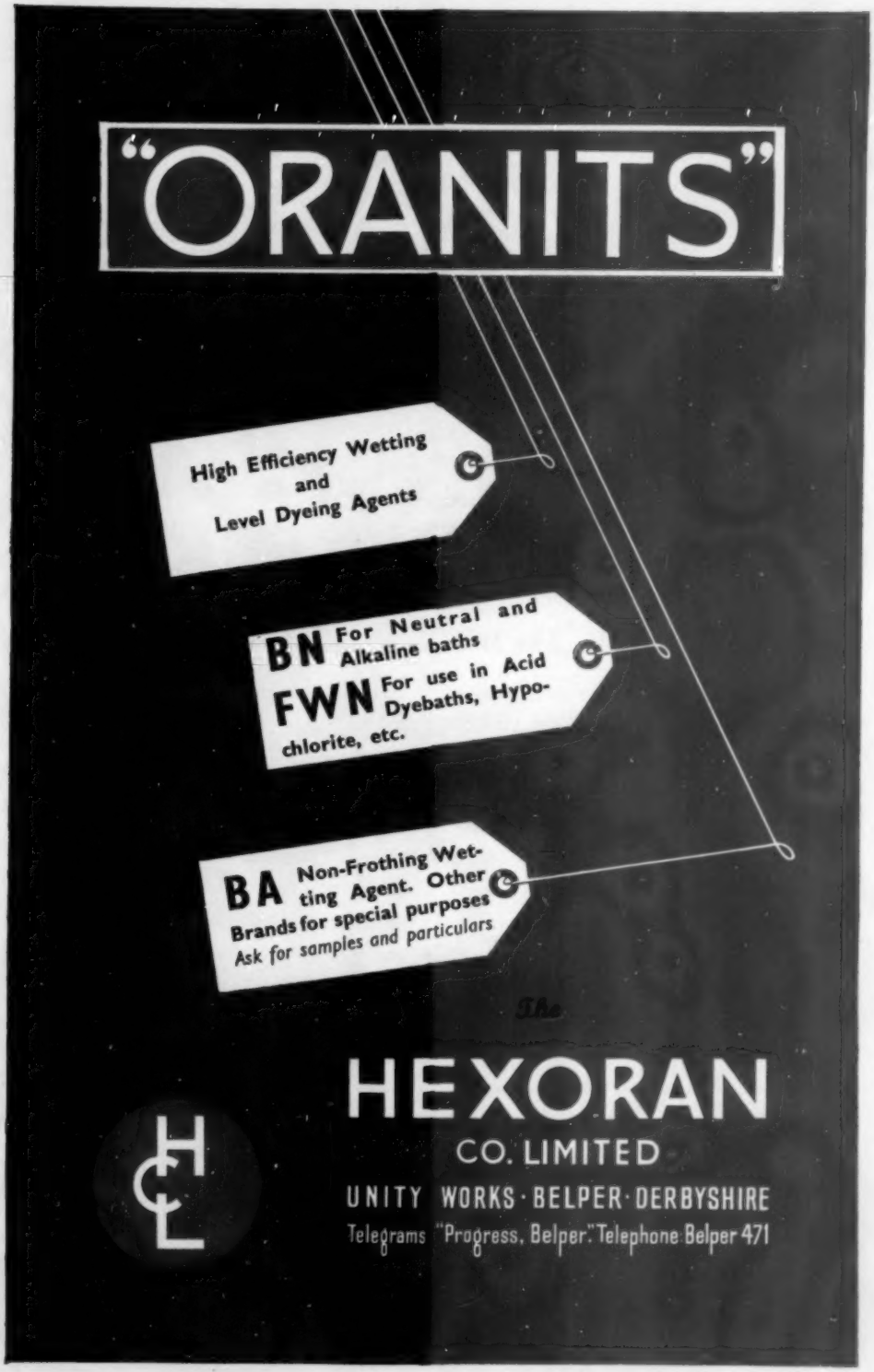
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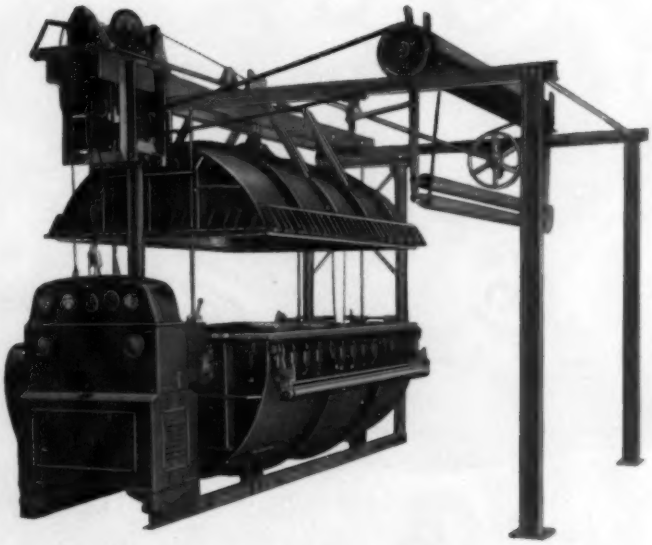

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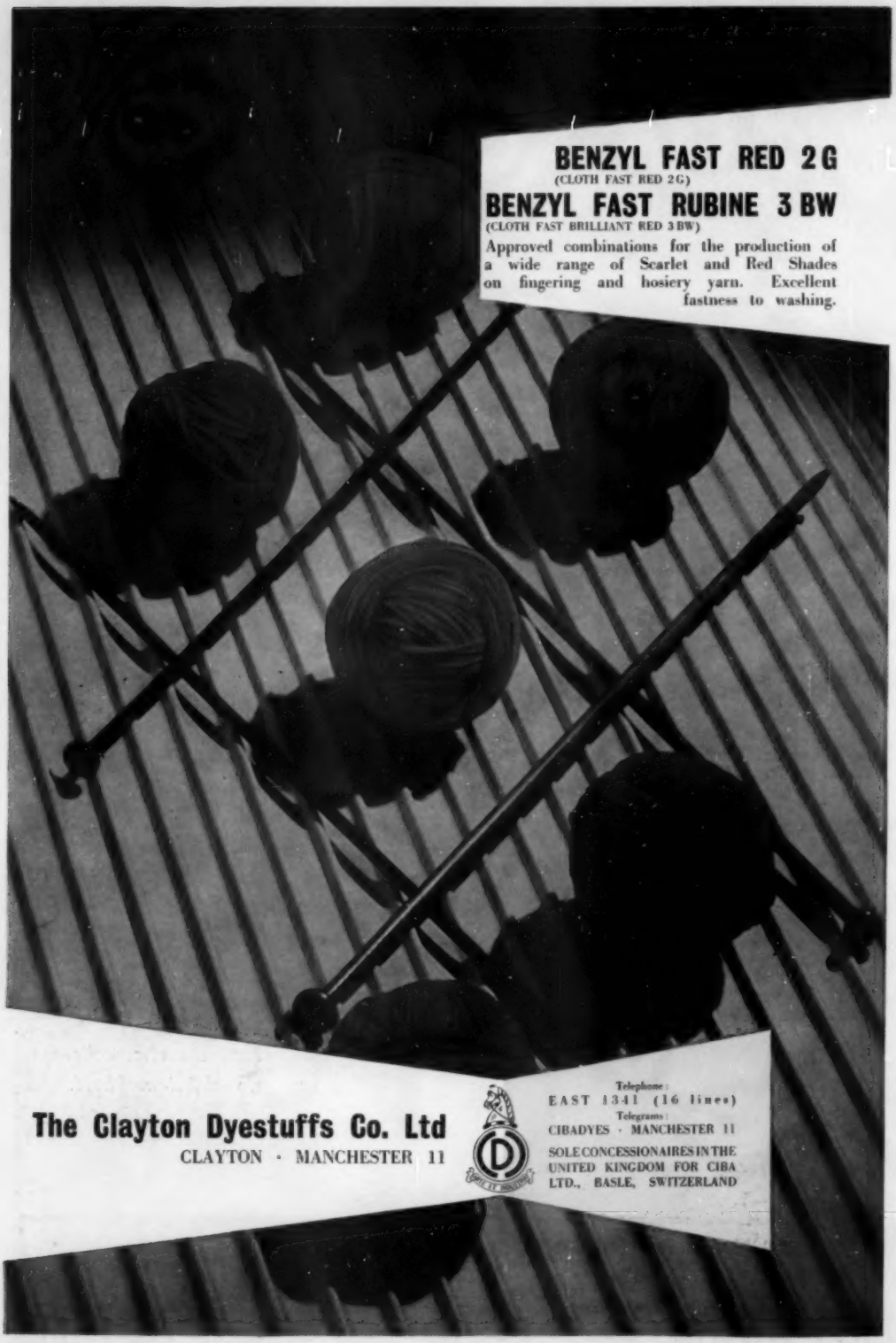
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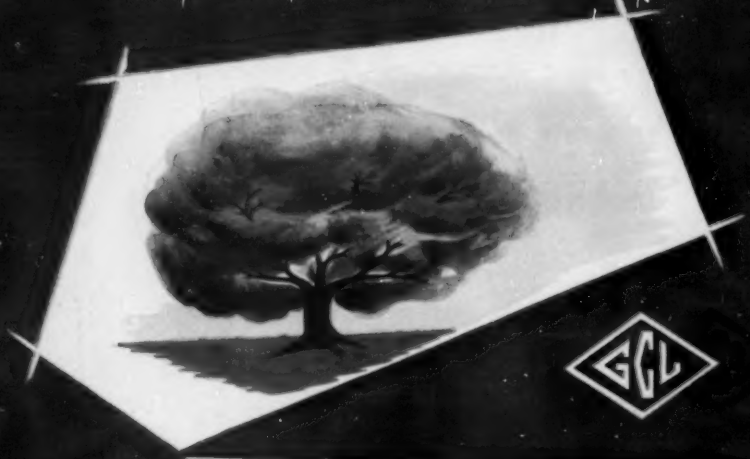

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A new and useful range of Cationic Softening Agents for all textile fibres

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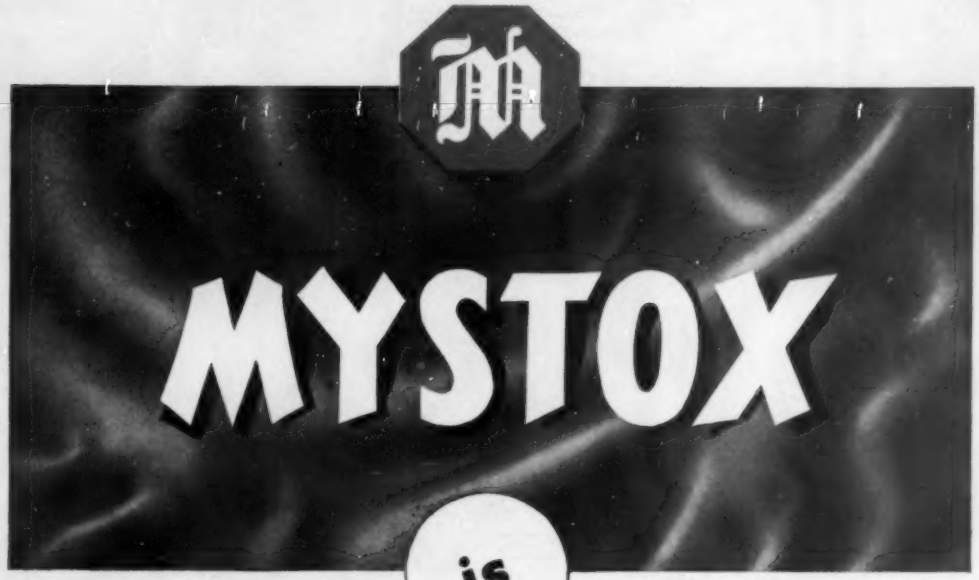

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FOR

WOOL

TOWN END

Town End Chemical Works Limited

BRAMLEY LEEDS

He piloted the firm through the war years and through the period of transition to scientific control of operations, at which point the firm reached new standards of production in fast-to-boiling dyeings. It may be of interest to recall that one customer had well over 800 standard shades. Intricate combinations of colours in ombre, colour, and white, and two-, three-, four-, five-, and six-colour effects in the same hank (all fast to boiling), were in regular production. During this period hand labour was replaced first by the Klauder-Weldon and later by the Gerber and similar machines.

G. T. Oldham maintained the tradition of public service, serving on Honley Urban District Council and acting as Chairman in 1925. He was appointed a J.P. in 1924 and became a West Riding County Councillor in 1928. He died in 1937.

Unfortunately, just when things seemed to be "set fair", the threat of Japanese competition, which had always been present but did not appear to be serious, suddenly assumed alarming proportions and reached a head about 1934, when a child's dress which had been dyed, woven, and made up in Japan, transported to this country, and imported, duty being paid, could be bought for less than the silk could be dyed here. Strenuous efforts to meet this competition were made, the bulk of the strain falling on George Hall Oldham, grandson of the founder of the firm, who had joined his father as a partner shortly after he had attained his majority. More and more machinery was installed in an attempt to decrease labour costs, but eventually he was compelled to close the family business. He died two years ago at the early age of 51.

All credit is due to the three generations of the Oldham family for their hard work, careful planning, and willingness to try out new ideas which brought them success and made the name of Oldham synonymous with all that was best in silk dyeing. The business found employment for between 100 and 120 employees, a number of whom worked there for over 50 years.

The collapse could be said to be due, in some measure, to the competition of synthetic fibres, but principally to world circumstances quite outside the control of the Oldham family.

APPENDIX

Prices taken from Contract Book

1903	Rhodamine B	1s.	3½d. per lb.
1903	Chrysophenine G	2s.	3d. per lb.
1903	Quinoline Yellow	1s.	11d. per lb.
1904	Milling Red G	2s.	0d. per lb.
1903	D.O.V.	65s.	0d. per ton
1913	Coal	10s.	10d. per ton
1915	Petroleum spirit	1s.	2d. per gal. including tax
1916	Petroleum spirit	2s.	5d. per gal. including tax
1936	Hydrosulphite		3½d. per lb.
1937	Hydrogen peroxide (100 vol.)		8½d. per lb.

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Theories of Dyeing

May I congratulate the Publications Committee on their excellent aim to make dyeing theory plain to the practical dyer: at the same time admire the boldness of H. A. Turner[1] and L. Peters[2] in tackling so difficult a project?

I would, however, like to ask the following questions—What standard of knowledge of physics and mathematics is required to understand the logarithmic equations, the constants, and the assumptions of mathematical physics in the two papers? How many of the practical dyer members of the Society, for whom the papers are written, have this requisite knowledge to understand the arguments? I venture to assert that the percentage is very low. H. A. Turner in his letter[3] says that dyeing theory "has gone fluid again". At a meeting of the West Riding Section held last winter, hydrogen bonding was described as bunk. Nobody in the large audience protested. What has the practical dyer member to make of it all? The answer is to concentrate on avoiding "cobblers". C. M. WHITTAKER

1 WEAPONNESS PARK
SCARBOROUGH
26th April 1955

[1] Turner, H. A., J.S.D.C., **71**, 29 (Jan.), 96 (Feb. 1955).
[2] Peters, L., J.S.D.C., **71**, 174 (April 1955).
[3] Turner, H. A., J.S.D.C., **71**, 181 (April 1955).

Testing Fastness to Laundering and Dry Cleaning

It was with particular interest that I read the letter by my friends Davies and Rhodes in the March issue of the *Journal*[1], and many will be grateful to them for it. I agree most heartily with their comments and, in fact, was about to write on that subject and other matters connected with dyers and colourists and the laundry and dry-cleaning industries.

How often do technologists forget that the dyes used on the goods they finish must be laundered or dry-cleaned at some time? And how rarely do they envisage the processes used by the industries under discussion and act accordingly, or advise action to their customers?. Surely it is now more important than ever that the dyer, printer, and finisher should know the end use to which the yarns, fabrics, or other goods he is commissioned to process are put. It would then be axiomatic to dye or print e.g. overall weaves with selected dyes, e.g. vats or azoics (selected) and, perhaps, sulphur dyes for cheaper qualities, and so on. An example of continued inappropriate finishing is the raised yellow dyed fabric used for making household dusters. Need I quote the dye used? Why is it not realised that such a duster will bleed

in a wash and mark off, and that such a mark-off cannot be removed? One is reminded of the comment by Scholefield at Blackpool that a smear of Benzopurpurine could not be removed. However good the sorting department of a laundry, and however many the classifications they sort into, the "yellow duster" will find its way also between "white work" with inevitable damage.

During the past fifteen years a great part of my interest has been with laundry and dry-cleaning establishments. Although both these industries, important as they are, have excellent research organisations, viz. the British Launderers Research Association and the Dyers and Cleaners Research Organisation, it is to be regretted that only a minority of firms are members of either or both. My experience has shown that the launderer or the cleaner is often blamed for faulty work when, in fact, the cause of the complaint lies elsewhere.

There can be no doubt whatever that the dye-makers should now extend the "fastness columns" of their excellent pattern cards to show the fastness of their dyes to laundering and dry cleaning. Considerable bulk work has shown that the "fastness to washing" figures now quoted do not suffice. It is not really remarkable that, for instance, all sulphur dyes will bleed in a three-wash process, i.e. two washes and a boil, all three in the presence of sodium metasilicate, sodium carbonate, or a mixture of the two, soap or detergent, and possibly, for some goods, a little sodium hydroxide.

I am well aware of the difficulties for the dye-makers in this matter, since, I submit, there is no reliable method in a laboratory to test detergency in the sense under discussion. I regard "artificial soils" (there seems to be a new one each week) as useless. Without any wish to become ribald I declare that no human being is known to me who exudes the oils used in such soils, nor does anyone living outside cities or large towns have much carbonaceous matter on his clothes. Firms well known to me have built laboratory washing machines to scale and washed articles to scale in them, but the tests proved useless. In any case the removal of "artificial soils" does not imply that household soils can be removed by the same recipe. Only large-scale work is of value, i.e. that in which the test piece (naturally soiled) is attached to some goods being laundered. Similarly, I submit that household goods are not bashed by rubber or steel balls in a washing machine, so "wash wheel" results are almost useless to a launderer. I have found "naturally" soiled test pieces simple to prepare and effective, but this is not the place to discuss that point.

Properly processed cutches and mineral khakis and most vat- and azoic-dyed goods will withstand severe laundering (and severe laundering is essential, e.g. on butchers' and fishmongers' aprons, hairdressers' overalls, and so forth), but not all by any means would be rated 5.

Fortunately, the difficulty arises only with cellulosic, regenerated cellulosic, and some synthetic fibres. The present fastness figures for proteins should satisfy.

In future, pattern cards should show the fastness of dyes to solvents, and on all substrates, including resins and plastics of all kinds. It is fortunate that, in this case, all fastnesses to solvents can be tested under laboratory conditions. The following are in question—(1) white spirit, (2) trichloroethylene, (3) tetrachloroethylene (perchlorethylene), and (4) distillate.

Owing to human carelessness and errors it will be necessary to quote "figures" under the normal cold and under warm conditions, as the temperature rises if uncontrolled at the end of runs in an afternoon. Technologists will know that many disperse dyes, azoics, and pigments may be seriously attacked (solubilised or changed in colour) by some solvents.

The comments made above are my own and do not necessarily coincide with the opinions of firms with which I may have been connected.

HERBERT À BRASSARD

136 GROVE HALL COURT
HALL ROAD
LONDON N.W.8

23rd April 1955

[1] Davies, E. J., and Rhodes, W. K., J.S.D.C., 71, 143 (March 1955).

The claim made by Mr. à Brassard that only large-scale testing is of value in determining such factors as launderability is shared by many others, including the writers, but we would certainly include dry cleanability among these factors and not rely on a laboratory test for solvent fastness. For instance, the Dyers and Cleaners Research Organisation, undertaking a considerable amount of test work for manufacturers of new and novel effects, does not rely upon laboratory test data for its conclusions, but rather uses them as a guide to the desirability and design of tests on a works scale. There are many examples which can be quoted, but the following may prove of interest.

Simple laboratory tests on flock prints involving the use of a dry-cleaning solvent in a wash-wheel or shaker apparatus may indicate a satisfactory performance level when the new fabric is employed. With fabrics having a pile type of surface, however, the mechanical action induced in such apparatus is quite different from that involved in a cleaning process where fabrics may slide one over the other. For this reason we have found it necessary to introduce a brushing technique, for which we employ trichloroethylene as the solvent, and then finally works tests are carried out in both white spirit and trichloroethylene. Even such tests are incomplete, since they do not take account of the effects of wear, which are most important for this type of fabric. Thus, new garments which can be described as dry-cleanable

in white spirit may show loss of flock in perspiration-wear friction areas when cleaned in the same solvent after normal wear. Thus, a dress will show a semicircular area across the neck and shoulders and patches on the upper arms where the adhesive bonding flock to fabric has deteriorated by the action of perspiration, warmth, and friction in wear. It is surprising how often the position of the partner's hand in dancing can be readily observed with adhesive-effect fabrics!

For such reasons, when testing unworn flock prints we have been led to the use of an empirical solvent-brushing technique in trichloroethylene which we find by experience to give a reasonable indication of the likely result in commercial dry cleaning after wear.

Finally, the time may be coming when testing in white spirit and perchlorethylene will be desirable, but at the moment the effects of white spirit and trichloroethylene require observation. Of these two, trichloroethylene is chemically the more active, and since the amount of dry cleaning undertaken in chlorinated solvents in this country is about 30% of the whole and is increasing, we, like other bodies, regard trichloroethylene as the test solvent for dry cleanability.

E. J. Davies
W. K. Rhodes

Dyers and Cleaners Research Organisation
4 Lifton Place
Leeds 2

24th May 1955

"Man-made"

The word "man-made" now seems to appear frequently in the *Journal*. On page 246 of the May issue it occurs three times. It is an ugly word.

Science and technology do, of course, give rise to ugly words, most of which are tolerated and often taken into common use because they add to our power of expression, or in some way are valuable. But, Sir, what advantage has this hyphenated little horror over the familiar and pleasant *artificial*?

J. S. Heaton

Tame House
Reddish Vale
Stockport
Cheshire

26th May 1955

Notes

Meetings of Council and Committees
June

Council— No meeting
Finance and General Purposes— 8th
Publications— 21st
Colour Index Editorial Panel— 16th
Fastness Tests Co-ordinating— 21st
Terms and Definitions— 20th
Perkin Organising— 10th
Perkin Executive— 10th
Northern Ireland Symposium— 18th
Review of Textile Progress— 24th

Death

We regret to report the loss by death of Mr. H. B. Briggs.

Reports of Sections for the Session 1954–1955

HUDDERSFIELD SECTION

Once again we have to report a successful session. Although we were compelled to change our place of meeting, this did not affect the attendance at meetings.

We held our annual dinner last October, with a record attendance. Our friends from the Midlands Section (Alf Carpenter and Sidney Meadows) came up and entertained us magnificently.

As in previous years, we held two joint lectures with outside bodies. The first, in November, when Dr. F. F. Elsworth spoke on *Chemistry in the Woollen Industry*, was held with the Huddersfield Textile Society. The second, with the Royal Institute of Chemistry, was on *Dyes for Uses other than Textiles*.

All lectures were of a high standard and of a practical nature, and we terminated our session with a lecture by one of our own members (Mr. H. Senior) preceded by the presentation of the records and documents of the firm of G. W. Oldham, handed to the President by Master George Oldham for safekeeping by the Society [see p. 399].

Our thanks are due to the Chairman and all members of the Committee, without whose help we could not have achieved such a successful session.

H. Turner
Honorary Secretary, Huddersfield Section

LONDON SECTION

This, the Jubilee session of the London Section, has been worthy of the event. Our technical meetings at the Royal Society Rooms have attracted record attendances, on three occasions numbering approximately 100; and the Third London Lecture was heard by nearly 200. A good start was made to the new membership campaign, twelve new members being elected during the year.

The lecture programme included joint meetings with the Textile Institute and with the Guild of Dyers and Cleaners. The latter was the first

occasion we had held a joint meeting with the Guild, and the former was the first post-war meeting held jointly with the Institute. Both these meetings were very successful and well attended.

The Third London Lecture was given by Mr. P. E. Jones, Deputy Keeper of Records to the Corporation of London, who dealt with *The Guilds of the City of London;* it maintained the high standard associated with this series. We were honoured to welcome the President of the Society at this meeting, and also the Prime Warden and the Renter Warden of the Worshipful Company of Dyers. It was a great pleasure also to have Mr. F. W. Walker, a Founder Member of the Section, with us.

A lecture meeting was held again at Luton, and as usual much appreciated by the local members.

Regarding the social programme, a Dinner Dance was again held at the Waldorf Hotel, and was probably the most successful yet, about 80 members and guests taking part. The programme concluded with a dinner following the Annual General Meeting.

H. W. TAYLOR
Honorary Secretary, London Section

MANCHESTER SECTION

Seven meetings have been held in Manchester this session and one joint meeting with the Textile Institute in Macclesfield. The external meeting in Macclesfield was particularly successful, with a very large attendance. There has been no general improvement in attendance at the normal meetings held in Manchester, although the average attendance has been good, owing mainly to the very large number of members and guests present at our Ladies' Evening.

This year a symposium has again been held, with the difference that it occupied the whole day instead of the afternoon and the evening, which had been the practice during previous years.

The Section's thanks are due to Imperial Chemical Industries Ltd. (Dyestuffs Division) for their hospitality in inviting the Section to visit their Dyehouse Laboratories for the Ladies' Evening. The tour of the laboratories and the subsequent supper and discussion provided an enjoyable and interesting evening for a large number of members and guests.

The session's meetings of the Junior Branch have been extremely successful, mainly owing to an increase in the attendance of the more junior members of the Society. This has resulted from Dr. J. M. Preston's agreement to the scheme whereby certain students taking evening classes in the Textile Chemistry Department attend the Junior Branch meetings as part of their course. During the past session, fifteen students have been present at each lecture under this scheme.

The thanks of the Section must be recorded to all those who have contributed to meetings and to Dr. Preston and the Manchester College of Technology for facilities for committee meetings. Although a further attempt was made to secure the Gas Showrooms for our meetings in Manchester, this was not successful, and the meetings have been held, once again, at the headquarters of the Textile Institute, to whom we are obliged for permission to use their lecture theatre.

The Annual Dance was held this year at the Lesser Free Trade Hall. Two hundred members and guests attended. The function was thoroughly enjoyed, but there was not complete satisfaction with the accommodation. It is evident that the question of venue for this function has not yet been satisfactorily solved.

The Knecht Memorial Prize for 1953–54 has been awarded to Mr. J. P. Critchley, who was a student in the Department of Applied Chemistry (Colouring Matters) at the Manchester College of Technology.

Mr. G. S. J. White, Chairman of the Section for the past two years, retired at the Annual General Meeting in April and was succeeded by Mr. J. Boulton, with Mr. J. W. Reidy as Vice-chairman. Mr. White has chaired both meetings of the Section and of the Committee with expert tact and with great enthusiasm for the Section and the Society. The Section is indebted to him for the great interest he has taken in its affairs.

Mr. F. Scholefield and Dr. C. M. Whittaker are to be congratulated on being elected Honorary Members of the Society; Mr. G. S. J. White is to be congratulated on the award to him of the Society's Gold Medal, and Mr. C. C. Wilcock on the award of the Silver Medal. Dr. H. A. Thomas is to be congratulated on the award of the Chardonnet Medal. The Section is proud that these awards have been made to present or past members.

During the past year the Section increased its membership from 622 to 652.

H. R. HADFIELD
Honorary Secretary, Manchester Section

MIDLANDS SECTION

During the session under review eight lecture meetings were sponsored by the Section (Leicester 2, Nottingham 2, Derby 1, Loughborough 3), while three further meetings were held jointly with textile associations and the Textile Institute in Hinckley, Leicester, and Kidderminster. As our support is drawn from a scattered area (50% of the audience in any centre has usually travelled 25 miles), the very bad weather conditions affected average attendance, which was down to 60. The standard of the lectures was well maintained. In addition, the Annual Section Dinner and the Ladies' Evening Dinner Dance attracted record support, while two works visits were well attended.

Increase in membership of the Section is well in line with that of the Society as a whole, the figures being—new members 15, transferred to the Section 7, transferred from the Section 5, resignation 1; net increase 16.

At the Annual General Meeting of the Section in April, congratulations and thanks were extended to Mr. S. R. Meadows on the conclusion of a very successful two years in the Chair. He is succeeded by Mr. C. A. Mills. From the committee Messrs. W. L. Langton, C. A. Mills, W. Penn, J. Saunders,

and J. Rayment retired by rota. The first four, together with Mr. T. E. Charlesworth, were elected for a further term of three years, while Mr. Rayment was elected to a casual vacancy for one year.

The Section has been happy to bear its part in the active work of the Society, and we would like to acknowledge the services on various committees of Messrs. W. A. Edwards, W. Penn, H. C. Olpin, A. P. Kershaw, and A. G. Tyler, the last two also being regular attenders of Council meetings. We would also like to congratulate the following—Mr. W. A. Edwards on once more being elected a Vice-president; Mr. A. S. Cluley on his election to Council; and the following Midlands members who have received the honour of Fellowship of the Society—Messrs. M. V. Campbell, A. J. I. Harding, W. Johnson, R. J. S. Linn, and R. J. Roberts.

A. W. CARPENTER
Honorary Secretary, Midlands Section

NORTHERN IRELAND SECTION

This session has been quietly successful in the respects that lecture attendances have been up to average and there has been a slight increase in membership.

The following is a summary of activities—

LECTURES—Seven lecture meetings were held. These dealt mainly with practical matters and were all well attended except one, which was affected by very adverse weather conditions.

COMMITTEES—Five committee meetings were held during this session. There were also five Symposium Committee meetings held for the purpose of dealing with social arrangements in connection with the Portrush Symposium.

ANNUAL DINNER—We had the pleasure of entertaining our President (Mr. Fred Smith), Senator H. Quin, Mr. H. Foster, and the General Secretary at this function, which was well attended, and which was presided over by our newly elected Chairman, Mr. R. G. J. Reid.

DINNER DANCE—We held our first Dinner Dance in February. There was an attendance of 186 people, and this function was so enjoyable that we propose making it an annual event.

GOLF COMPETITION—We had an attendance of 32 at our second annual Golf Competition, which was regarded as a successful function.

This Section feels highly honoured that Council have decided to hold a Symposium at Portrush this year, and we shall spare no efforts to see that the social events are successful. We are in process of arranging bus tours to well known beauty spots, golf competitions for ladies and gentlemen, and formal and informal dances.

JAMES PORTER
Honorary Secretary, Northern Ireland Section

SCOTTISH SECTION

We are pleased to report a very successful and enjoyable session, during the course of which seven meetings were held, all in Glasgow. Attendances have been remarkably good, and arrangements have been made for a somewhat larger room for next session.

The Ladies' Evening, which was held in February, was very successful and the Committee are greatly encouraged by the good support given to this function.

Membership of the Section again shows an increase, the total figures for the past three years being—

March 1953 ...	198
March 1954 ...	210
March 1955 ...	224

In January we had a visit from Mr. C. O. Clark, who gave a talk and clarified points in connection with the Diplomas Scheme. Mr. Clark also visited the Paisley Technical College and addressed a meeting of staff and students on this matter.

During April a very well supported three-day Conference was held in the Royal Technical College, Glasgow, on "Fibres, Plastics, and Colours". Our Section, together with local sections of the Textile Institute and the Plastics Institute, collaborated with the College authorities in arranging this.

At the Annual General Meeting of the Section Messrs. A. R. Edge, W. G. B. Grant, D. B. F. McAndrew, and G. Miller retired from the Committee by rota. All were eligible for re-election, and, as no other nominations had been received, they were duly elected.

Arrangements are well in hand for the 1955–56 session, and the Committee hope to present a programme of practical papers which will appeal to all members.

F. ATACK
Honorary Secretary, Scottish Section

WEST RIDING SECTION

The Section under the Chairmanship of Mr. G. B. Angus has continued its usual activities.

The two social events—the Supper and Smoker and the Ladies' Evening, arranged by Mr. J. M. Goodall (Honorary Social Secretary) and Mr. R. Hullah—were extremely successful. The popularity of these events increases each year.

Nine lecture meetings were arranged, seven to be held in Bradford and two to be held in Leeds. Unfortunately, owing to the illness of the lecturer, the first Leeds meeting had to be cancelled at short notice.

Included in the meetings at Bradford was a non-technical lecture, on *Oriental Printing Methods and Prints*, to which ladies were invited. The attendance at this function raises the question of the desirability of holding future meetings of this nature.

Also included in the activities of the Section was a talk by Mr. C. O. Clark (Convener of the

Diplomas Executive Subcommittee) on the Diplomas Scheme of the Society. Unfortunately, bad weather influenced attendance at this important discussion.

The Dyers and Finishers Association's donation of £10 10s. 0d. was used this year for an essay competition, open as before to all Junior Members of the West Riding Section. Six papers were submitted, and the following three awards were made—

First Prize to Mr. J. H. Clegg, for his paper on *Identification of Disperse Dyes on Nylon*

Second Prize to Mr. G. A. Holmebakk, for his paper on *A Study of the Dyeing of Terylene Polyester Fibre and Courpleta Triacetate Rayon*

Third Prize to Mr. N. G. Morton, for his paper on *The Production and Dyeing of Wool–Modified Wool Mixtures with special reference to Metal-complex Dyes.*

The assessors of the papers stated in their report that the standard of all the papers was excellent, and they considered that the competition was useful training for the Junior Members.

The Committee met on four occasions.

G. E. Styan
Honorary Secretary, West Riding Section

BRADFORD JUNIOR BRANCH

The activities of the Bradford Junior Branch have been well maintained during the past year. A total of nine meetings in all have been held, of which one took the form of a film evening. The average attendance has been 43 members. Two works visits were arranged, the numbers attending being limited to thirty in each case. The West Riding Section Junior Members' Competition was this year held as a written competition, and attracted six entries. It will be run on similar lines during the coming year.

On the social side, the Annual Dance was held in February. About 230 people attended, and the function was both a financial and a social success.

The Branch is in a flourishing state and assisting in no small way to further the interests of the Society as a whole. Thanks are due for the continued support of members, without which it would be impossible to maintain the activities of the Branch.

N. G. Morton
Honorary Secretary, Bradford Junior Branch

Dr. A. B. D. Cassie, C.B.E.

The award of the C.B.E. to Dr. A. B. D. Cassie, Director of Research of the Wool Industries Research Association, was announced in the Birthday Honours List.

Jubilee of the London Section

As mentioned in the sessional report (p. 403), 1954–1955 was the Jubilee Session of the London Section. Unfortunately, the old minutes book of the Section was lost or destroyed during the bombing of London during the 1939–1945 war, but the following brief account of the foundation of the Section is based on notes written from memory by Mr. F. W. Walker and on the contemporary report in the *Journal*.

Fifty years ago a few members of the Society were located in the London area, but were attached to either the West Riding or the Manchester Section. One of them was Morris Charles Lamb, who had recently come from the Leather Industries Department of Leeds University (then the Yorkshire College) to assume the post of Lecturer in Leather Dyeing and Dressing at Herold's Institute, Bermondsey.

Lamb was really the Founder of the London Section, was its first Honorary Secretary, and afterwards Chairman. The first meeting was held in a lecture room at Herold's Institute on 24th June 1904. Here a Sectional Committee was formed representing dye suppliers and dye users, and Sir Thomas Devereux Pile was in the chair.

Sir Thomas was at that time interested in the firm of Brooke, Simpson, & Spiller, the old-established dye manufacturers of Hackney Wick. He had gathered together a small team, including Dr. J. C. Cain and Dr. E. Feilmann, with Mr. H. Wilkinson in charge of the dyeing laboratories. Prof. A. G. Green was consultant. Some of the staff of B. S. & S. were amongst the early London members.

The lectures in those early days had a pronounced bias towards leather processing, which is understandable, as Lamb and his colleagues gave quite a few lectures to the Section. Later a recruiting campaign for new members developed, and the Section gathered its membership from dyers and cleaners, fur dyers, felt and straw hat dyers, fabric printers, hosiery dyers, colour photography, and a host of small dye-consuming industries in and around London. One of the troubles of those days was to obtain lectures of sufficient interest to the majority of the members and not merely to attract the faithful few.

The meeting places were about as numerous and as diverse as the industries represented. The problem was to find suitable rooms sufficiently central to gather good attendances. Special meetings were sometimes held in hotels, but ordinary meetings were at the Leathersellers Technical College, the Borough Polytechnic, Birkbeck Bank Chambers, King's College, the Criterion Restaurant, and the Chemical Society's rooms among others.

A gentleman who devoted much time and energy to the Section was Frank J. Farrell, whose lecture on "Dry Dyeing" aroused a great deal of interest at the time and gave a fillip to the dyeing and cleaning membership. Others were Dr. J. C. Cain, Dr. E. Feilmann, Mr. W. P. Dreaper, Mr. James Blair, Dr. C. Dorée, and Mr. R. E. Crowther of Kodak, to mention only a few.

The impact of two world wars, in which London suffered more than most cities, had its effect in slowing the growth of the London Section, and at one time the Section was "ticking over" and no more.

A further stage in development was reached when the Worshipful Company of Dyers allowed the use of its ancient hall for the Section meetings, which were held for some years on the first Friday of the month, and indeed still are. With the growth in membership thanks to the work of the Committee, it became apparent that a more central meeting place must be found. It would appear that the Society and the Section owe much to Dr. H. W. Ellis for carrying out the negotiations with the Royal Society for the use of their splendidly appointed rooms.

A lecture that comes to mind of the not too distant days was given at a joint meeting of the London Section and the Textile Institute in the Hall of the Barrett Street Trade Schools at the rear of Selfridges. Here a packed meeting (many were seated in gangways and on window ledges) heard a lecture by Dr. C. M. Whittaker on *Viscose Rayon Dyeing*, a subject on which he was, of course, very much at home. There was a lively discussion afterwards. The outstanding lesson of this meeting was that, given the right man and the right subject, as good attendances can be expected in London as in the industrial North. F. W. W.

British Rayon Research Association

The new buildings of the British Rayon Research Association at Heald Green, Wythenshawe, Manchester 22, were formally opened by H.R.H. The Duke of Edinburgh on 11th May 1955. The buildings include laboratories for fundamental and applied research, technology sheds (including a dyehouse), a library, and a well equipped lecture theatre to seat 250. The work of the B.R.R.A. is mainly concerned with problems relating to the use of rayon. Investigations in the dyeing, printing, and finishing sections are being undertaken into the processing of fabric made from blended yarns, package drying, delustring of cellulose acetate, the mechanism of gas fume fading, weathering of acetate rayon, resin finishing of viscose rayon cloths, and fugitive tints. Fundamental research is concerned with the physics and chemistry of cellulose, application of flash photolysis to fading and phototendering, the mechanism of the uptake of dyes by cellulose acetate, and the absorption of vaporised dyes by hydrophobic fibres.

National Federation of Dyers and Cleaners

The Federation consists of three associations, in London and the South, the Northern and Midland counties, and Scotland respectively. Although the original objects were to regulate prices and wages, the main activities are now concerned with public relations, technical training, and research (Dyers and Cleaners Research Organisation). At an "at home" held in the Dyers' Hall, London E.C.4, on 12th May 1955, to celebrate the Jubilee of the Incorporated Association of London and Southern Dyers and Cleaners, Mr. A. J. Crockatt (Chairman of the Manufacturers Contact Committee) described some aspects of the work of the D.C.R.O. In many cases, e.g. interlinings and flock prints, manufacturers have been persuaded to adjust their methods to render their products "dry-cleanable"; in others, e.g. cellulose triacetate, it would be necessary for dry cleaners to alter their methods.

Leeds Technical Library

A new Library of Commerce, Science, and Technology, containing over 60,000 volumes, was formally opened in the Central Library, Leeds, by Sir Philip Morris (President of the Library Association and Vice-chancellor of Bristol University) on 27th April 1955.

Protein Fibres from Feathers

A large surplus of chicken feathers is available in the U.S.A., and attempts are being made to utilise this by-product in various ways. It has been found that a simple extraction process using alcohol and water dissolves ca. 70% of the feather protein, which can be recovered as a powder. Fibres can be made by extruding the solvent-moistened powder at high temperatures. The highly stretched fibres have rather better dry strength than wool, but poor wet strength. Even after treatment with acid–formaldehyde the wet strength is only approximately half the dry strength. All known methods for extracting the protein, however, are too costly for general economic production, and in addition the pigments in the feathers are intimately associated with the protein and are carried through into the fibres, so that any fibre production would be restricted to white feathers. (*Ind. Eng. Chem.*, **47**, 13A (March 1955).) W. K. R.

Metallic Yarns

Metallic yarns are being manufactured in the U.S.A. under the names *Lurex*, *Metlon*, *Reynolds aluminum yarn*, and *Lamé* from aluminium foil 0·00045 in. thick. A special thermoplastic adhesive containing dyes or pigments is applied to each side of the foil, and after heat-treatment a sheet of transparent cellulose acetate–butyrate is laminated to each side under high pressure. The laminated material is split into filaments $\frac{1}{110}$ or even $\frac{1}{210}$ in. wide, normally $\frac{1}{64}$ in., and wound on to spools. The yarn is mainly used along with a supporting yarn of cotton, rayon, etc. Laminated acetate rayon yarns can be washed up to 160°F. and can be dry-cleaned in solvents suitable for acetate rayon. A new development is the use of Mylar (polyethylene terephthalate) film in place of cellulose acetate. The Mylar laminated yarn has relatively high strength, which allows it to be used without a supporting thread, and it can be processed at the boil. The improved properties are expected to result in new and more widespread applications for metallic yarns. (*Ind. Eng. Chem.*, **47**, 11A–13A (March 1955).) W. K. R.

OBITUARY NOTICES

Emil Adolf Krähenbühl

World War II delayed by five years my first meeting with one who, following subsequent events, I would have felt privileged to have met earlier in my life, namely Emil Krähenbühl. He joined the Society of Chemical Industry in Basle—now Ciba Ltd.—in 1941. It was not until 1946 that I was able to revisit Switzerland, renew my acquaintance with that hospitable country, and meet, for the first time, Emil Krähenbühl in his capacity of an Assistant Manager and Chief Colourist of Ciba Ltd.

I shall always regret those five years which delayed this occasion, since, when I had the opportunity of meeting him, I felt an immediate appreciation that here was a capable chemist and technician, and one with a thorough understanding of the requirements of industry, in which we both had similar interests.

I would, perhaps, be failing in a duty if I did not draw special attention to his wide knowledge of printing. On this subject Emil Krähenbühl was an authority, and few have equalled him.

Our common interest gave rise to a personal and family friendship, the memory of which I and my family will treasure for all times. Emil Krähenbühl was not only a gifted scientist and technician, but was equally rich and generous in his ability to give a lasting and trustworthy friendship.

Emil Krähenbühl visited this country on many occasions, and it is safe to say, from my personal knowledge of him, that apart from his beloved Switzerland there was no country which he appreciated more. He loved our countryside, and when opportunity permitted would travel far to view one of our historic buildings. Our cultural and educational interest always impressed him. He was a member of our Society, and was particularly proud that he personally had been privileged to present papers before us.

He has contributed in no small measure information for the Second Edition of the *Colour Index* on behalf of the Swiss dyemaking firms. The care taken in compiling the information for this publication and its accuracy were characteristic of Emil Krähenbühl's ability to deal with detail in a most thorough way. The Chairman and the Editorial Panel of the *Colour Index* will always be grateful to him for his valued contributions.

It was a fitting reward for his efforts on behalf of the Society and for his international high reputation as a chemist and colourist that the Society honoured him with its Fellowship. He greatly appreciated this distinction.

The world is poorer without him. His death in February 1955 at the relatively early age of 57 is a great loss.

To his family all who knew him extend their deep sympathy, and trust that they will be comforted by many happy memories of the past, with the knowledge that those who knew him best will remember him the longest.

JAMES G. GRUNDY

J. Campbell Gray

It is always a sad occasion when an obituary comes to be written of a member of the Society. And those of his era, and many others who met him, will have heard with regret of the death of J. Campbell Gray. The term "a dour Highlander" is generally a gross exaggeration and does not fit by any means the majority of those kindly if serious-minded people. But Gray, although he had much charm of manner, was of the dour type, and preferred not to wear his heart upon his sleeve.

The Society's and my good friend James R. Hannay has most kindly reminded me of the 1890s era, when Campbell Gray's uncle was the manager of the Strines Printworks. At that time Campbell Gray was a student at the Manchester College of Technology under the delightful Dr. E. Knecht.

Around 1909 Gray became the Honorary Secretary (under Pennington and Knecht) of the Manchester Section of the Society; in fact, the Section was organised under their guidance. Despite Gray's extreme reserve there were many friendly streaks in his character, and he always aimed to introduce new members to their colleagues and to lecturers. It is not without historical interest to recollect that in those days discussions after a lecture were considered taboo. It now seems strange that members of the Society had "secrets" not open to technical discussion. Gray thought comments would not be in the best taste, although Knecht never hesitated to make his comments. It can be said that Hannay did much to evolve a new tradition, and gradually Gray's objections were overcome.

There is no doubt at all that Gray was a most assiduous and efficient secretary of the Manchester Section, and such enthusiasm as he permitted himself to have was given fully to colleagues and members of the Society.

When he left Lancashire, he almost lost all interest in calico printing. He would occasionally attend meetings of the London Section, but preferred to live a hermit-like life. Even when working for Klingers in London, where he was provided with a small laboratory, he preferred to work alone. It must be emphasised, however, that, despite this extreme reserve, he maintained a definite charm of manner.

It can be said that Gray was a conscientious worker and not without special capabilities.

But let it be repeated that there is no doubt whatever that the Society owes his memory considerable gratitude for long and faithful service to its Manchester Section. HERBERT A BRASSARD

New Books and Publications

Chemisorption

By B. M. W. Trapnell. Pp. vii + 265. London: Butterworths Scientific Publications 1955. Price, 35s. 0d.

The reviewer has some sympathy with the author's wife, who, according to the preface, finds the subject of chemisorption incomparably dull. Certainly this cannot be classed as an exciting book, but nevertheless the author is to be congratulated on collecting together such a large amount of material, much of it previously available only in original papers. In these days of rapidly accumulating scientific literature this is a necessary and most valuable service.

As the name somewhat inelegantly suggests, chemisorption is a process of adsorption in which the substance adsorbed is held to the surface by chemical bonds rather than by physical forces of attraction. After describing methods and techniques of measurement, the author deals with experimental results and theoretical ideas concerning velocities of adsorption and desorption—activation energies and velocity constants—equilibrium adsorption isotherms, heats of adsorption, mechanisms of chemisorption, and the mobility of adsorbed layers. Two final chapters aim to show the relationship between chemisorption and heterogeneous catalysis.

There is something in this book for all who are interested in the general subject of adsorption.

J. Crank

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Badische Anilin- und Soda-Fabrik

Helizarine Colours— This card contains a range of 20 pigment colours for textile printing. They are fixed upon the fibre by direct printing in conjunction with an oil-in-water emulsion of a resin binder followed by heating at 120–140°c. for 3–5 min. Washing-off or soaping after printing is not necessary. They are recommended for machine and screen printing on practically all types of fibres and fibre mixtures. The prints have good fastness to light, very good fastness to dry and wet rubbing, chlorine, and washing, and a soft handle. The Helizarine colours may also be used alongside azoic combinations and aniline black, as coloured resists under Variamine Blue and aniline black, and in coloured discharges.

Helizarine Colours in Pad–Dyeing Processes— With a few exceptions the pigment colours described in the previous card are all suitable for pad–dyeing on cloth, the resin binder, Helizarine Binder FD, being incorporated as an aqueous dispersion in the padding liquor together with Textapret C new, an acrylic acid copolymer, as a stabiliser, Condensol A, an accelerator for the resin binder, and Tylose DKL. They can be applied to almost all types of textile materials, including fibre mixtures, to give dyeings of very good fastness to light and wet treatments and having a soft handle. Fixation is carried out at 130–140°c. for 4–5 min. Simultaneous production of either a crease-resist or an embossed finish is possible. Pale to medium-depth dyeings only are recommended, as if heavy dyeings are produced the amount of resin binder required adversely affects the handle of the cloth.

Ortolan Dyestuffs— This card contains dyeings in three depths of six Ortolan dyes and sixteen mixture dyeings. They are metal-complex dyes recommended for dyeing wool in all forms, silk, and polyamide fibres to give dyeings of excellent fastness to light and wet treatments. They are applied from weakly acid solution but also have good affinity from a neutral dyebath. Uniperol W is recommended as a levelling agent, and with it shading may be carried out at high temperature. In the absence of this agent, it is suggested, dyeing should be carried out in the presence of ammonium acetate only, and the dyebath cooled to 60°c. before shading dye is added. A notable feature is the high light fastness of very pale dyeings, e.g. Ortolan Yellow G, $\frac{1}{12}$ of standard depth, 6. They are also of interest for direct and discharge printing on wool, silk, and polyamide fibres and for Vigoureux printing.

Indanthren Blue CLR— This vat dye gives reddish blues on cellulosic fibres. It is recommended for use alone and in mixtures for dyeing all types of material where maximum fastness to light and wet treatments is demanded. It is also of interest on silk. Type 8059 and Colloisol brands are available for pigmentation and continuous dyeing techniques. Fastness figures on cotton include— Light 7–8, soda boiling 3–4, chlorine (*b*) 4.

Indanthren Cyanine B— This vat dye gives bright greenish blues on cotton and linen, the hue on mercerised cotton and regenerated cellulose rayon being progressively redder. It is particularly recommended as the main component for navies fast to iron and water-spotting on raincoat poplins. Type 8059 is available for pigment padding processes, Type 8015 for vulcanising, and a Colloisol brand for continuous dyeing processes. Fastness figures on cotton include— Light 6, soda boiling (*b*) 3–4, chlorine (*b*) 4–5.

Indanthren Grey GG Colloisol— This vat dye is of particular interest for producing by pigment padding neutral greys of excellent fastness to light and wet treatments on cotton and linen cloth. The fine state of subdivision of the particles and consequent rapidity of reduction make it very suitable for continuous dyeing processes. Fastness figures on cotton include— Light 7–8, washing (*c*) 4–5, chlorine (*b*) 3.

Cassella Farbwerke Mainkur

Immedial Pure Green FFG— This sulphur dye gives bright yellowish greens on cotton and regenerated cellulose rayons. Dyeings have very good wet fastness, and their fastness to light is improved by aftertreatment with potassium dichromate–copper sulphate, although they become bluer in colour. They are unaffected by crease-resist finishing. Immedial Pure Green FFG is suitable for package dyeing, and a Type 8015 is available free from copper and manganese. Fastness figures for a dyeing on cotton aftertreated with dichromate and copper include— Light 4, washing (95°c.) 3–4.

Cassurit MLP Liquid— This is a melamine–formaldehyde precondensate marketed as a viscous solution which may be diluted with cold or lukewarm water without free formaldehyde being developed.

Ciba Ltd.

ORASOL AND ORACET DYES—This card describes a range of 24 Orasol dyes and 14 Oracet dyes, which are shown incorporated in cellulose acetate film. They are soluble in many organic media and are free from diluents. They are recommended for colouring polymethacrylate, polystyrene, polyvinyl chloride, cellulose acetate, and celluloid moulding compositions by incorporation before or during moulding, and may also be used for dyeing finished plastics articles from solvent solutions.

DYES FOR SUÈDE LEATHER—Dyeings on full chrome suède calf of 66 dyes are included in this card as follows—18 Fast Leather (acid) dyes, 19 acid dyes, 19 Neolan dyes, and ten Chrome Leather (mordant) dyes. Details of suitable dyeing methods and fastness data are appended.

CIBACET BRILLIANT SCARLET RG—This disperse dye gives orange-scarlets on acetate rayon and polyamide fibres, dyeings on the latter being particularly bright, and on both fibres the fastness to light, wet treatments, and burnt gas fumes is good. It is also suitable for direct and discharge printing. Fastness figures on acetate rayon include—Light 6, washing (ECE*a*) 3, burnt gas fumes (AATCC) 5.

CIBANOGENE BORDEAUX R; BL—These two stabilised diazo compound–Cibanaphthol printing compositions fill the colour gap between Cibanogene Red R and Violet B. Prints may be developed in an acid bath, by acid steaming, or in neutral steam by a special recipe. They may be printed alongside a wide range of other types of dye and are also suitable for coloured resists under Cibantine dyes and aniline black. Fastness figures on cotton include—*Cibanogene Bordeaux R:* Light 4–5, washing 4–5, chlorine (ECE*a*) 4–5. *Cibanogene Bordeaux BL:* Light 5, washing 5, chlorine (ECE*c*) 4.

CIBANOGENE NAVY BLUE B; BLACK B—These also are stabilised diazo compound–Cibanaphthol compositions for printing cellulosic fibres. Prints are developed by either acid steam or neutral steam in the presence of Cibanogene Developer S. Wet development is possible only with Navy Blue B, and in this case weaker prints are obtained. Fastness figures include—*Cibanogene Navy Blue B:* Light 5, washing 5, chlorine (ECE*a*) 5. *Cibanogene Black B:* Light 5–6, washing 5, chlorine (ECE*a*) 5.

CIBAKONE YELLOW LGR—This vat dye gives reddish yellows of excellent fastness to light and wet treatments on cellulosic fibres and is particularly recommended for coloured woven goods and furnishings. Solid dyeings are obtained on cotton–viscose rayon mixtures. Light fastness is unaffected by crease-resist finishing and only a very slight change in hue occurs. Fastness figures on cotton include—Light 6–7, soda boiling (ECE*a*) plus chlorine (ECE*b*) 4–5.

COPRANTEX B—This circular describes the properties and uses of a copper-containing cationic auxiliary product designed for aftertreating dyeings of ordinary direct and aftercopperable direct dyes to improve their fastness to washing, water, and perspiration. It may be applied in conjunction with synthetic resin finishes and softening agents, e.g. Sapamines KW, WL, and WP. The circular contains aftertreated dyeings on cotton of 37 dyes together with the corresponding direct dyeings for comparison of hue, and a full range of fastness data for both sets of dyeings is included. A supplement gives light fastness figures for the same range dyed on viscose rayon staple cloth.

LYOFIX AS—A finishing agent for giving weighted, filled, and stiff finishes fast to washing on cellulosic materials.

PHOBOTEX FT—This product, based on thermosetting resins, is recommended for producing permanent water-repellent finishes on cellulosic fibres, acetate rayon, silk, polyamide fibres, and Orlon.

SAPAMINE OC—This is a cationic auxiliary product particularly recommended as a softening agent for Orlon. It is also of interest on cellulosic fibres, acetate rayon, silk, wool, and polyamide fibres. It has hardly any effect on the hue and light fastness of dyeings, and the softening is unimpaired by high-temperature setting up to 215°C.

Clayton Dyestuffs Co. Ltd.

RENAMING AND REGROUPING OF ACID WOOL DYES—The nomenclature of the acid wool dyes has been standardised, so that now in all cases the group name gives a clear indication of the dyeing characteristics of the dye. There are five groups—Kiton Fast and Kiton dyes are applied from a sulphuric acid bath to give dyeings having a light fastness of 4–5 or better or below 4–5, respectively. The Benzyl Fast and Alizarine Fast and Benzyl dyes are dyed under less strongly acid conditions, the designation "Fast" again denoting that the light fastness is 4–5 or better. The Cloth Fast Blues are normally applied in the presence of ammonium salts; the name "Cloth Fast" is retained only for the blues. The booklet lists the dyes in groups according to their new generic names with the former name alongside, and also lists both new and old names in alphabetical order with the respective old or new name alongside in each case.

CIBACET RUBINE R—This homogeneous disperse dye gives bluish reds on acetate rayon and polyamide fibres, the hue on the latter being almost violet. It is also suitable for dyeing cellulose triacetate and withstands permanent pleating. It is suitable for direct printing on acetate rayon and nylon, and dyeings on both of these fibres are dischargeable to white. Fastness figures on acetate rayon include—Light 6, washing 4–5, sublimation 4–5.

KITON FAST BLUE SRL—This acid dye is suitable for use alone and in mixtures for dyeing all forms of wool goods, and may be applied at 108°C. without significant alteration in hue. Although the colour becomes redder and duller if nitrites are present in the dyebath, this tendency can be overcome by adding sulphamic acid. It is suitable for dyeing polyamide fibres, but medium depths only are obtainable. Fastness figures on wool include—Light 5–6, carbonising 4–5, perspiration 2–3.

Compagnie Française des Matières Colorantes

THE DYEING OF COTTON AND VISCOSE RAYON WITH DIRECT DYES: VOL. 1—DIRECT DYEING—This card contains dyeings in two depths on delustred viscose rayon crêpe and cotton sateen of 180 direct dyes grouped as follows—61 Diazol Light dyes distinguished by the good fastness to light and 119 Diazol and other dyes. Certain members of the latter group carry the additional designation "Brilliant" or "Fast", indicating that they are distinguished by their brightness or slightly superior fastness to a particular agency. Details of pretreatments, dyeing methods, aftertreatment with Fixogene TN, selection of dyes for specific purposes, and fastness properties are included in 22 pages of text.

PRINTS ON VISCOSE RAYON WITH ACID AND DIRECT DYES—This card contains direct prints of 33 direct and 19 acid dyes. They have been produced by the urea process on matt viscose rayon crêpe.

Farbenfabriken Bayer

GALLOCHROME YELLOW 5GL: GALLOCHROME YELLOW RRL—These are chrome dyes for printing cellulosic fibres, and give greenish and reddish yellows, respectively, having good fastness to light in medium and full depths. Fixation is by steaming for 10 min., and consequently they are suitable for printing on naphtholated grounds alongside colour salts and diazotised bases. They may also be used for coloured discharges. Fastness figures on cotton include—*Yellow 5GL:* Light 5–6, washing (*c*) (95°C.) 3. *Yellow RRL:* Light 5, washing (*c*) (95°C.) 3.

INDANTHREN "POWDER SUPERFINE" DYESTUFFS; CIRCULARS NO. LE 356E AND LE 389E—These circulars, the second of which is a supplement to the first, list 10 Indanthren vat dyes now available together with Indanthren Red F3B as Powder Superfine brands. The first circular gives details of their physical and dyeing characteristics. They are non-dusting and are readily wetted to give aqueous dispersions of complete stability and of considerably smaller particle size than the Type 8059 brands. They are particularly recommended for dyeing cloth and yarn by pigmentation methods.

RAPIDOGEN BORDEAUX N–1RB 54—Bright, deep, bluish reds, of very good fastness to light and washing, are obtained by direct printing with this stabilised azoic

combination. It can be developed by either neutral or acid steaming or by a wet steaming process; addition of Rapidogen Salt A or Rapidogen Developer N is unnecessary. Subject to final confirmation, prints of not less than the indicated, prescribed minimum depth on general-purpose textiles or on fabrics to be repeatedly laundered may carry the Indanthren fastness label. Fastness figures on cotton include— Light 4–5, washing (*c*) 5, soda boiling 5, chlorine 5.

SIRIUS SUPRA GREY GN-L— This homogeneous direct dye gives bluish greys on all forms of cellulosic fibres. It is also of interest for printing. The fastness to wet treatments of dyeings and prints is considerably improved by aftertreatment with Levogen WW. With Type 8000 acetate rayon is unstained, and a Type 8015 is available for dyeing material which is subsequently to be rubber-proofed. Dyeings may be discharged to a good white under both neutral and alkaline conditions, Type 8002 being recommended for such work. Fastness figures on cotton include— Light 5–6; washing (*a*) (40°C.) 2, aftertreated dyeing 4–5; perspiration 1–2, aftertreated dyeing 5.

BENZO CUPREN B— This is a cation-active copper-containing auxiliary product for aftercoppering a range of 18 copperable dyes introduced as the Benzo Cupren dyes, as well as most of the Benzo Cuprol (Benzo Fast Copper) dyes. It is marketed as a blue liquid which can be diluted with water to any desired extent. The card contains dyeings on cotton and viscose rayon including a range of untreated dyeings to indicate the change in hue which results. Aftertreated dyeings have good fastness to light and wet treatments, including mild washing, but will not withstand repeated severe washing, soda boiling, or chlorine, and are unsuitable for vulcanising.

BENZO CUPROL RED FBL (BENZO FAST COPPER RED FBL)— This homogeneous direct dye gives bright reds of good fastness to light and wet treatments on cellulosic fibres when aftertreated with Benzo Fast Copper Salt, Benzo Cupren B, or copper sulphate. It is also of interest on silk and for direct prints on cotton and regenerated cellulosic rayons, for which purpose a concentrated brand is available. Dyeings are dischargeable under alkaline conditions if anthraquinone is added to the discharge paste. Fastness figures for a dyeing on cotton aftertreated with copper sulphate include— Light 6, washing (*b*) (60°C.) 4, perspiration 4–5.

INDANTHREN BRILLIANT PINK BK— This vat dye gives bright bluish pinks on cellulosic fibres. Dyeings on curtain and furnishing fabrics and on textile materials generally may be marked with the Indanthren fastness label irrespective of the depth of the dyeing. The powder superfine brand is suitable for pigmentation dyeing methods. It is suitable for silk dyeing. Dyed grounds may discharge white with Rongalite CL, and it is also of interest for direct printing. Fastness figures on cotton include— Light 7, washing (*c*) (100°C.) 4, chlorine (*b*) 5.

PERLIT C PROCESS— This leaflet describes a method of producing a waterproof finish on cellulosic materials which shows good resistance to dry-cleaning solvents.

ISOLAN DYES— A range of metal-complex dyes applied to all forms of wool goods from very weakly acid solutions (pH 5–6·5). Dyeings of excellent fastness to light and wet treatments are obtained, a feature being the high light fastness of even pale colours, e.g. Isolan Bordeaux RLS, ¼ standard depth 6–7. Four dyes are described in this card, which includes dyeings in five depths on cloth, and 43 mixture dyeings on slubbing, yarn, and woollen and worsted material. They are useful for union dyeing and Vigoureux printing and also for dyeing silk.

SUPRANOL BRILLIANT RED 3BW— This acid dye gives very bright bluish reds of good fastness to light and wet treatments when dyed on wool from a weakly acid dyebath. It is also of interest for dyeing the wool in wool–cellulosic fibre mixtures and silk. Fastness figures on wool include— Light 5, washing (*b*) (60°C.) 3–4, carbonising 4–5.

Geigy Co. Ltd.

DYESTUFFS FOR WOOL PRINTING— Seventy-nine direct machine prints on chlorinated wool are included in this card. They have been produced with selected fast-to-milling acid dyes, neutral-dyeing metal-complex dyes, level-dyeing acid dyes, and direct dyes.

Imperial Chemical Industries Ltd.

DYESTUFFS FOR ACETATE RAYON–CELLULOSIC UNIONS — This card contains dyeings of 22 disperse dyes and 30 direct dyes on a 50 : 50 acetate rayon–viscose rayon brocade. The direct dyes are of CR quality, which gives little or no staining on acetate rayon. A dyeing of Chlorazol Black LFS diazotised and developed with 2:3-hydroxynaphthoic acid is also included, since, although not available in CR quality, it is recommended for use in conjunction with Dispersol Diazo Black B for producing solid blacks. Light fastness figures are included for dyeings on both bright and titania-delustred rayon, and attention is drawn to dyes which are particularly suitable for dyeing materials which are subsequently to be given a crease-resist finish. Notes in the text include one on the rapid fading of dyeings of certain direct dyes on bright, and particularly on pigmented, viscose rayon when exposed to light in a damp condition; attention is also drawn to the catalytic action of certain yellows, e.g. CR Durazol Yellow G, GR, and 3R, in accelerating the fading of certain other direct dyes when wet or damp mixture dyeings are exposed.

NERAN BRILLIANT DYESTUFFS FOR LEATHER FINISHING — A range of 13 dyes are illustrated on card sprayed with a solution containing 2% of dye and 12·5% of Neran Glazing Finish A. This range of water-soluble dyes have been developed for incorporation in aqueous pigment finishes to increase their brightness. Brilliance, high tinctorial value, high solubility, and low salt content are essential together with resistance to the alkali present in the finish and the formaldehyde customarily used for improving the rubbing fastness of the final product.

HEXAGON DIGEST NO. 20— The contents of this issue include *Discharge Printing on Natural Silk— Colorimetric Estimation of the Discharge Properties of Dyes* by E. Cowgill; *The Absorption of Copper and Manganese by Acetate Rayon, Nylon, and Terylene Polyester Fibre* by J. G. Graham and I. M. S. Walls; and *Investigations into the Dyeing of Hydrophobic Fibres. II— The Use of Dyeing Assistants* by T. Vickerstaff. The third paper suggests that the function of both the soluble and the insoluble type of dyeing assistant is to loosen the molecular structure of the film and so increase the rate of dyeing. The use of an assistant can be equivalent to a 10–20°c. rise in dyeing temperature.

Sandoz Ltd.

THE SANDOCRYL PROCESS FOR THE DYEING OF POLYACRYLONITRILE FIBRES— This circular describes a proprietary method for applying acid dyes to polyacrylonitrile fibres based on the cuprous ion technique. Metallic copper replaces the more usual reducing agents— hydroxylamine sulphate, sodium bisulphite, or glyoxal. In the presence of copper sulphate under acid conditions cuprous ions are produced—

$$Cu + Cu^{++} \rightarrow 2\,Cu^{+}$$

It is claimed that the risks of too rapid absorption of dye and consequent unlevel dyeing, possible reduction of dye, and precipitation of metallic copper are eliminated. Dyeing is carried out from a sulphuric acid solution at pH 2 in the presence of sufficient copper wire or sheet to give an effective surface area of 100 sq. cm. per litre of dye solution and 1–1½ times as much copper sulphate as dye. Dyeing is started at 85–90°c., the temperature raised to not less than 95°c., and dyeing continued at 95°c. until exhaustion is complete. It is advantageous if dyeing can be carried out above 100°c., when the amounts of copper and copper sulphate required are smaller. A range of 13 Sandocryl C–1 dyes, shown dyed in three depths on Orlon 42 cloth, is recommended for dyeing polyacrylonitrile fibres by this method to give dyeings of very high fastness to light and wet treatments. Fastness figures for Sandocryl Black BLC–1 include— Light 8 (Fade-Ometer), alkaline milling 4.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Pulverising Materials. FBy. *BP* 729,596

The materials are placed in a container and subjected to the exploding action of gases which have been detonated, preferably in a combustion chamber of the container, the pulverised materials being passed off with the combustion gases and the scavenging gases. C. O. C.

Thread-storing, Thread-advancing Device. Studi e Brevetti Applicazioni Tessili. *BP* 728,414

Applying Liquid to a Running Thread or Yarn. British Enka. *BP* 728,745

Thread or yarn, in the course of passing at speed from one spool to another, changes direction by passing helically round the surface of a vertical tube. Liquid for treating the yarn is pumped up the inside of the tube under careful control and emerges from the top against an overlapping cap which makes it flow down the outside surface, thus making contact with the yarn. Conditions can be arranged to treat yarns travelling at up to 500 yd. per min. with up to 100% uptake of liquid. J. W. B.

Aftertreatment of Artificial Silk Threads. Vereinigte Glanzstoff-Fabriken. *BP* 729,467

Artificial silk threads pass through an aftertreatment bath in spaced parallel relationship and are withdrawn from the exit end in such a manner that adjacent threads have different emergent angles. This is effected by leading the threads over a grooved roll, adjacent grooves being of different depths, or over a notched bar, adjacent notches being of different depths, or by being guided over a roll so that alternate threads pass over and under the roll. J. W. B.

Fluid Treatment of Cakes or Hollow Packages of Thread. Société de la Viscose Suisse. *BP* 729,197

Method and apparatus in which the column of packages is closed by a lid, the bearing (sealing) pressure of which is always approximately proportional to the pressure of the treating fluid within the column and is never more or less than slightly in excess of that pressure. C. O. C.

Winding and Unwinding Tyre Ply Stock. Firestone Tire & Rubber Co. *BP* 728,465

Regularising Tension of Artificial Threads during Liquid Treatments. Vereinigte Glanzstoff-Fabriken. *BP* 729,178

Thread tension in liquid aftertreatment baths is regularised, while maintaining a minimum tension value, by varying the guide rollers. If one particular bath causes swelling, splaying-out of the filaments, and consequent uneven running, the diameter of the roller between this bath and the next is increased slightly, e.g. by 1%, above that of the roller which preceded the first bath. This causes tautening and steadier travel, and the diameters of subsequent intermediate guide rollers are similarly adjusted according to swelling effects of the individual baths. J. W. B.

Dye Vat equipped with Stock Solution Distributing and Mixing Means. J. De Marco. *USP* 2,676,478

Controlling the Tension in a Length of Material between Winding and Unwinding Rolls. General Electric Co. *BP* 728,492

Large Metal Rolls. Bowaters Development & Research. *BP* 728,148

A form of construction of large rolls, e.g. length 12 ft., external diameter 18 in.–4 ft., internal diameter 8 in.–2 ft., which prevents the shell breaking away should it fracture. C. O. C.

Slasher Rolls for Sizing Textile Yarns. Dayton Rubber Co. *BP* 729,191

Slasher rolls with greatly improved life comprise a surface layer of natural or synthetic rubber which contains a substantial proportion of wool, e.g. 1–2 parts by wt. of wool, to 1 part of rubber, the wool fibres having a length of $\frac{1}{16}$ in.–$\frac{1}{4}$ in. J. W. B.

Screen Printing Machine. J. N. McLaurin. *BP* 729,674

The squeegee is used as a carrier for colour during the return stroke, the squeegee picking up colour at the end of the printing stroke, holding it during the non-printing stroke and depositing the colour on to the screen before the printing stroke. C. O. C.

Compositions for making Printing Rollers. Perma-Flex Industries. *BP* 728,732

A core is surrounded by a layer of a composition made up of insoluble polyvinyl chloride and/or vinyl chloride-acetate copolymers containing > 5% of acetate and linear polyesters of sebacic and/or adipic acid with dihydroxy paraffinic alcohols of 3–8 C and not more than 1 C atom between the C atoms to which the OH groups are attached, a plasticiser and as tackifier a rosin, rosin-modified phenolic resin, or modified rosin. The composition must have hardness of 10–30 measured by a Shore Durometer. C. O. C.

Apparatus for Coating Paper. Champion Paper & Fibre Co. *USP* 2,676,563

Apparatus for forming on an applicator roll for transfer to the paper, a film of coating material of controlled thickness throughout. C. O. C.

Printing Machines Primarily for Printing Transfers for Pottery. Service (Engineers). *BP* 729,562

The paper is fed between the printing roller and a resilient roller. The printing roller is hollow and carried by a pair of flanged collets or bosses, one fitting into each end of the roller, their diameter being not greater than that of the roller. The printing roller is driven from one end and the collet or boss supporting the other end is axially adjustable. This provides a machine having a simple and inexpensive mode of adjustment to different lengths or diameters of printing roller. C. O. C.

Garment Press. American Laundry Machinery Co. *BP* 729,977

A press in which a low pressure is applied immediately it is closed but has means whereby the pressure can be increased while the press remains closed. C. O. C.

Embossing Rolls for Use in making Reflex-reflecting Strips or Sheets. Grote Manufacturing Co. *BP* 728,722

Embossing rolls having optically-smooth cavities are produced by passing clean, fire-polished glass beads into the nip of two rotating metal rolls, where one of the rolls, that to be embossed, is polished and softer than the other, the pressure roller. With the use of sufficient pressure the glass beads are pressed into the softer roller forming hemispherical cavities in it having the same highly polished surface as the glass beads. The method described is suitable for rolls of large diameter and gives greater assurance of clean, well-formed embossing cavities, i.e. reduces the chances of fracture of some of the glass beads. C. O. C.

Machines for Shrinking Hat Bats. United States Hat Machinery Corpn. *BP* 728,860

A machine through which the bats are automatically fed so that they are returned from the exit to the entrance thereof, the angular position of the bats being automatically changed each time that they are introduced to the entrance of the machine. C. O. C.

Drying and Conditioning Leather. J. Kahn and M. Pier. *BP* 729,145

The skin is fastened to a supporting plate and then placed in air at 350–550°F. while simultaneously subjecting the skin to infrared radiation after which it is led to a second zone where it is treated with a current of air at a temperature less than that of the air in the first zone. C. O. C.

Coating Paper. Brightwater Paper Co. and Millers Falls Paper Co. *USP* 2,676,119

A method and machine for coating paper in which the necessary thickness of coating is applied in a single pass of the paper. C. O. C.

Dyeing Plastic Sheets for Use in Glare Screens. Libbey-Owens-Ford Glass Co. *BP* 729,755

A method of producing a laminated safety glass antiglare screen having a dyed area of gradually decreasing hue from an edge to the middle. Apparatus for dyeing plastic sheets for incorporation in such screens is described. C. O. C.

Electrostatic Application of Liquid Coating Materials. Licentia Patent-Verwaltungs-G.m.b.H. *BP* 729,842

Machine for Applying Circles or Bands of Colour around Cylindrical Objects such as Gelatin Capsules. R. P. Scherer Corpn. *BP* 728,517

Multi-colour Screen Printing of the Exterior of Cylinders, Cones or other Objects having an External Surface of Revolution. L. G. Dubuit. *BP* 729,105

Presses for Carrying Out Marking Operations. Masson Seeley & Co. *BP* 728,391

A press for marking ribbons, etc. by transfer of pigment, dye or the like from foil to the ribbon with or without application of heat. C. O. C.

II— WATER AND EFFLUENTS

Bibliography on Textile Waste. AATCC Piedmont Section. *Amer. Dyestuff Rep.*, **44**, P 168–P 190 (14 March 1955).

Continuous Ion Exchange with an Endless Belt of Phosphorylated Cotton. C. H. Muendel and W. A. Selke. *Ind. Eng. Chem.*, **47**, 374–379 (March 1955).

Phosphorylated cotton has a total ion-exchange capacity of the same order as commercial ion-exchange resins, but is unsuitable for use in acid systems owing to the hydrolysis of the cotton. Studies of the rate of exchange of phosphorylated cotton have shown that resistance to internal diffusion is the primary rate-limiting mechanism. A continuous counter-current ion-exchanger was constructed, based on an endless belt of phosphorylated cotton towelling, and was used successfully to concentrate copper solutions with brine regeneration. It is considered that the method could be brought to the same level of operation as obtains with resins. W. K. R.

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Long-chain Alkylbenzenesulphonates. F. W. Gray, J. F. Gerecht, and I. J. Krems. *J. Org. Chem.*, **20**, 511–524 (April 1955).

1-Phenyloctane, 1-phenyldodecane, 2-phenyltridecane, and 5-phenyldodecane have been prepared and sulphonated. The isomeric sulphonates were separated and identified. The *ortho* sulphonates have lower surface tension values, are better wetting agents, and give greater foam heights than the corresponding *para* isomers, but the *para* compounds are superior to the *ortho* deriv. in detergent properties. H. E. N.

Ultraviolet Radiation Absorbers. C. M. Knowles and S. R. Buc. *Proc. Chem. Specialities Mfrs. Assocn.*, 156–159 (May 1953): *Chem. Abs.*, **49**, 4408 (10 April 1955).

2 : 4-Dihydroxybenzophenone (I) and 2 : 2′-dihydroxy-4 : 4′-dimethoxybenzophenone (II) effectively absorb ultraviolet radiation without being destroyed by the absorbed energy. II is yellow whereas I is colourless. They are both similar in solubility and compatibility with plastics. They can be used as optical filters for protecting materials from ultraviolet radiation, e.g. lacquers, or as stabilisers for plastics and the dyes used in cosmetics. C. O. C.

PATENTS

Alkylated Aryl Sulphonate Detergents. Shell Development Co. *USP* 2,676,185

Method of preparing salts of alkyl aryl hydrocarbon sulphonates (Alk of > 11C) by a combination of carefully coordinated sulphonating and neutralising conditions which leads to the least formation of byproducts. C. O. C.

Surface Active Agents. British Petroleum Co. *BP* 728,433

Surface active agents are produced by treating a long chain alkyl hydroperoxide and/or an alkyl substituted naphthene hydroperoxide with SO_2 in presence of an aqueous and/or alcoholic medium and enough of a base to convert the hydroperoxide to a neutral salt of an alkyl hydrogen sulphate and/or a neutral salt of an alkyl substituted naphthene hydrogen sulphate.

BP 728,504

Salts of aryl substituted alkyl hydrogen sulphates, suitable as surface active agents, are produced by treating an aryl substituted alkyl hydroperoxide (Alk of > 3C, the hydroperoxide group is attached to a C atom which is not directly attached to the aromatic nucleus) with SO_2 or a sulphite and converting the resulting sulphate to a salt by treatment with a basic compound. C. O. C.

Concentrated Aqueous Soap Solution of Low Viscosity. Celanese Corpn. of America. *USP* 2,676,152

The viscosity of aqueous solutions containing > 15% soap have their viscosity reduced to a minimum by adding 2–10% (on the wt. of soap) each of a mineral oil and an emulsifying agent. When the soap is an oleate a similar reduction in viscosity is obtained by similar addition of an alkyl oleate (Alk of 3–10C). C. O. C.

Washing Powders. Metallgesellschaft. *BP* 728,582

When mixtures of synthetic detergents, carbonates and/or sulphates and/or phosphates and/or pyrophosphates and/or silicates are used on textiles in hard water the calcium and magnesium salts precipitated are adsorbed by the fibres where they form an incrustation and impart a dirty grey appearance. This effect can be avoided by incorporating with the detergent mixture a non-lime-stable mono- and/or diester of orthophosphoric acid containing at least one hydrocarbon radical of > 7 C and a chain of formula $O\cdot(RO)_n$ (R = aliphatic divalent radical of > 1 C; n = > 1), e.g. mono(*iso*octylphenyl)ester of orthophosphoric acid. C. O. C.

Spinning Lubricant. Metallgesellschaft. *BP* 728,748

Easily removable spinning lubricant comprises an aqueous emulsion of a substantially water insoluble neutral tertiary organic ester of orthophosphoric acid or a diester of an organic-substituted phosphonic acid, which contains at least one surface-active group having at least 6 C atoms in the molecule. It may be blended with conventional lubricants, e.g. equal parts of olein and a tertiary ester of orthophosphoric acid with oleyloctapolyglycolether, or a homogenised mixture of 40 parts (by wt.) of oleic acid, mineral oil (25), neutral tertiary ester of orthophosphoric acid with oleyldiglycol (10), secondary ester of orthophosphoric acid with lauryldiglycol ether (15) and water (10). J. W. B.

Textile Lubricant. Celanese Corpn. of America. *USP* 2,676,924

Staple fibres of an organic derivative of cellulose are satisfactorily lubricated and conditioned for spinning by treating them with a mixture of a partial mixed alkyl phosphate, an alkylolamine ester and/or amide of a long chain aliphatic acid, a tertiary amine, e.g. diethylcyclohexylamine, an alkyl phenol, e.g. diamyl phenol, and a mineral oil. C. O. C.

Polymerised Starch Esters and Ethers. National Starch Products. *BP* 729,302

A water dispersible starch derivative is prepared and its water dispersibility is then reduced, by reacting the starch in an aqueous alkaline medium with an amount of an organic anhydride, a chloroformate, an organic halide etherifying agent or an epoxy compound each containing the polymerisable olefinic group—

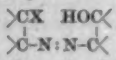
$$-\mathrm{C}=\mathrm{CH}_2$$
$$\quad\;\;\mathrm{R}$$

(R = H or Alk), so as to produce an ether or ester containing not more than 1 polymerisable olefinic radical per 15 anhydroglucose units. This is then brought into contact with a water soluble, free radical producing, vinyl

polymerisation type catalyst, e.g. $NaHSO_3$ or H_2O_2, perhaps with a catalyst activator, e.g. $Fe_2(SO_4)_3$, which reduces the water dispersibility of the starch derivative. Coated articles may be stiffened by applying the catalyst treatment subsequent to the starch impregnation, and will then retain their stiffness and crispness even after wet treatment. J. W. B.

Aldehyde-Guanylmelamine Condensates. Ciba. *BP* 730,018

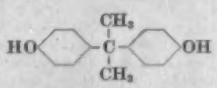
The condensates of mono-, di- or triguanylmelamines, with an aldehyde and the salts obtained by treating them with a water-soluble salt or of an amine in presence of a solvent, are used for improving the wet fastness properties of direct dyeings produced with dyes containing sulphonic or carboxylic acid groups. Used in conjunction with a copper salt they are especially advantageous for use with dyes containing the group—



(X = OH or COOH; the pair of C atoms bound to X belong to a cyclic diazo component, the pair of C atoms to which the OH is bound are those of any desired coupling component). C. O. C.

Fire-retardant Coating Compositions. Monsanto. *USP* 2,676,162

Potentially intumescing fire-retardant coating compositions for use on paper, wood, plastics, metal, etc., comprise a reaction product of anhydrous ammonia and phosphoryl chloride, a thermosetting resin, a film-forming condensate of—



with epichlorohydrin, and an organic vehicle including an organic solvent for the condensate. They air dry at room temperature to give coatings of good adhesion. C. O. C.

Stabilised Papain. Sterling Drug. *USP* 2,676,138

Incorporating into the fresh papain latex 0·1–3·0% of Na_2SO_3 before drying yields a product which retains its high proteolytic activity even when kept for a long time. The effect is enhanced if in addition to the sulphite a little of a phenolic compound, e.g. thymol, is also incorporated. C. O. C.

Plasticised Acrylonitrile Polymers. American Cyanamid Co. *USP* 2,676,161

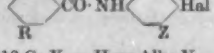
Cyanomethyl formate and di-(cyanomethyl) carbonate are solvents and plasticisers for polymers and copolymers of acrylonitrile. C. O. C.

Solvents and Swelling Agents for Acrylonitrile Polymers. BrC. *BP* 728,674

Acyloxy aliphatic nitriles of < 6 C, are good swelling agents or solvents for acrylonitrile polymers, e.g. formyloxyacetonitrile is a very good solvent and β-formyloxypropionitrile a very strong swelling agent for polyacrylonitrile. C. O. C.

Salicylanilides as Antiseptics. Gy. *BP* 728,098

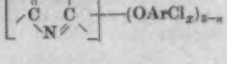
Compounds of formula—



(R = Alk of 4–12 C; X = H or Alk; Y = H or Hal; Z = H, Hal or trifluoromethyl), e.g. 5-*tert.* amyl salicylic acid-4′-chloroanilide, are good antiseptic agents for use on textiles to which they impart good resistance to the attacks of fungi and bacteria. C. O. C.

Fungicides. Ethyl Corpn. *BP* 728,675

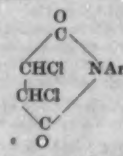
Compounds of formula—



(n = 1 or 2; x = 0–4) are excellent fungicides, some of which, e.g. 2:4-dichloro-6-(4-chlorophenoxy)-triazine dissolved in *cyclo*hexanone, and then diluted with kerosene, are suitable for application to textiles, leather, etc.

BP 728,994

Compounds of formula—



e.g. *N*-phenyl-*αα′*-dichlorosuccinimide, have similar use.

BP 729,448

2-Chloro-4:6-(diarylamino)-1:3:5-triazines or 2:4-dichloro-6-(arylamino)-1:3:5-triazines, have similar use. C. O. C.

Mordants for fixing Dyes in Photographic Layers. Eastman Kodak Co. *USP* 2,675,316

The effectiveness of basic mordants in preventing dye diffusion depends upon (1) degree of ionisation, the higher this is the less mordant is needed, (2) size, the mordant-dye salt formed must be too large to diffuse through the colloid, (3) solubility, the mordant must be dispersible in water and compatible with the colloid used. Polymers of formula—

$$R^1NR^2R^3$$

(R^1 = a polymeric structure; R^2 and R^3 = same or different, Alk or alicyclic or together = atoms to complete a saturated heterocyclic ring), e.g. polydiethylaminoethylmethacrylate, meet these requirements. C. O. C.

Copolymers of Organic Derivatives of Titanium and Silicon as Water-repellent Agents for Textiles. Monsanto. *BP* 728,751

A mixture of (*a*) a monomeric organic derivative of Ti, the organic groups being linked to Ti through O or N atoms, e.g. a tetra alkylorthotitanate, and (*b*) a monomeric Si compound of formula—

$$Si(OR)_nR_{4-n}$$

(R = monovalent organic radical, n = 1–4), e.g. tetraethylorthosilicate, is treated in an organic solvent with less water than is required for complete hydrolysis. The products are applied to textiles from anhydrous solution, the solvent removed and the treated textile exposed to a moist atmosphere. The moisture reacts with some of the remaining hydrolysable groups in the copolymer and the resultant coating then adheres strongly to the fibres. C. O. C.

Synthetic Tanning Agents (XII p. 430).
Synthetic Tannins (XII p. 430).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Mechanism of Aromatic Sulphonation and Sulphone Formation. W. H. C. Rueggeberg, T. W. Sauls, and S. L. Norwood. *J. Org. Chem.*, **20**, 455–465 (April 1955).

The reaction of benzene with SO_3 in liquid SO_2 produces the sulphonic acid in high yield together with a smaller amount of diphenyl sulphone. The amount of the sulphone formed increases with the temp. at which the reaction is carried out, but is decreased greatly by the presence of small amounts of acetic acid. It is concluded that probably the reaction proceeds via benzenepyrosulphonic acid $C_6H_5{\cdot}SO_2{\cdot}O{\cdot}SO_2{\cdot}OH$. H. E. N.

***iso*Propylcresols.** M. S. Carpenter and W. M. Easter. *J. Org. Chem.*, **20**, 401–411 (April 1955).

Effect of Layered Water on the Colour Reaction of Benzidine and Similar Compounds with Montmorillonite. H. Takahashi. *Bull. Chem. Soc. Japan*, **28**, 3–9 (Jan. 1955).

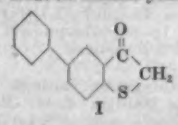
Influence of the Addition of High-molecular Electrolyte upon the Absorption Spectra and Fluorescence of Organic Dyes—IV. N. Mataga and M. Koizumi. *Bull. Chem. Soc. Japan*, **28**, 51–54 (Jan. 1955).

Changes in the intensity of fluorescence and the absorption spectra in the range 400–550 mμ. of aqueous soln. of uranin, eosin, and erythrosin ($1–2 \times 10^{-5}$ M.) on the addition of chitosan hydrochloride, glucosamine hydrochloride, and methylamine ($0–10^{-2}$ M.) have been measured. With uranin, the absorption spectrum changes markedly with addition of all three amines. The intensity of fluorescence decreases. With eosin and erythrosin, there is little change in either spectrum or fluorescence on addition of $CH_3{\cdot}NH_2$, but marked changes (decrease followed by recovery) with the others. A. J.

Nitration of 2-Aminothiazoles. J. B. Dickey, E. B. Towne, and G. F. Wright. *J. Org. Chem.*, **20**, 499–510 (April 1955).

Indigoid Dyes. XII—Thioindigoid Dyes derived from Diphenylmonosulphonic Acid. P. C. Dutta and D. Mandal. *J. Indian Chem. Soc.*, **31**, 827–831 (Nov. 1954).

5-Phenylthioindoxyl (I) is prepared from *p*-diphenylthioglycollic acid (itself prepared from diphenyl-4-sulphonic acid via the corresponding sulphonyl chloride and thiol) by the action of PCl_5 to give the acid chloride followed by ring closure with anhydrous $AlCl_3$. I could not be isolated in the pure state owing to atmospheric oxidation. From I the following dyes were prepared—(*a*) 2:2′-Bis-5-phenylthionaphthalene-indigo, by the action of $K_3Fe(CN)_6$ on a soln. of I in NaOH. m.p. > 290°c., dyes cotton pinkish violet from a yellow vat. (*b*) 2-(5-Phenylthionaphthalene)-3′-indole-indigo, by the action of HCl on a soln. of I and isatin in acetic acid. m.p. > 290°c., violet colour with conc. H_2SO_4, dyes cotton pinkish red from a yellow vat. (*c*) 2-(5-Phenylthionaphthen)-3′-(6′-bromo)-indole-indigo, as for (*b*) from *m*-bromoisatin. m.p. > 305°c., green with conc. H_2SO_4, dyes cotton deep violet–red from a yellow vat. (*d*) 2-(5-Phenylthionaphthen)-2′-, (5′ or 6′-nitro)-, (5′:6′-dinitro)- and (6′ or 5′-bromo)-acenaphthylene-indigo, as for (*b*) from 5-phenylthioindoxyl and the corresponding acenaphthenequinone derivatives. They show the following characteristics—Unsubst.: m.p. 243–244°c., deep green with conc. H_2SO_4, slightly sol. in alkaline hydrosulphite, dyeing cotton light pink; mononitro: m.p. 154°c., deep green with conc. H_2SO_4, dyes cotton greenish violet from an intense yellow vat; dinitro: m.p. 264°c., brown with conc. H_2SO_4, dyes cotton grey from an intense yellow vat; monobromo: m.p. 269°c., green with conc. H_2SO_4, slightly sol. in alkaline hydrosulphite, dyeing cotton light pink. (*e*) 2-(5-Phenylthionaphthen)-9′-phenanthrene-indigo, as for (*b*) from I and phenanthraquinone, m.p. 252°c., brownish green with conc. H_2SO_4, dyes cotton pale violet from pale yellow vat. (*f*) 2-(5-Phenylthioindoxyl)-1′-acenanthrenone, as for (*b*) from I and acenanthraquinone, m.p. > 310°c., brown with conc. H_2SO_4, almost insol. in alkaline hydrosulphite giving a very light pink on cotton. (*g*) Bis-5-phenylthionaphthen-ethylene-indigo, as for (*b*) from I and glyoxal sodium bisulphite, m.p. > 305°c., violet with conc. H_2SO_4, dyes cotton deep violet from an intense yellow vat.

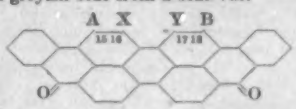


I

A. J.

Vat Dyes of the Benzanthrone Series. XXIX—Chlorination of 16:17-Dihydroxyviolanthrone with Phosphorus Pentachloride. T. Maki and M. Maezawa. *Bull. Chem. Soc. Japan*, **28**, 77–80 (Jan. 1955).

The action of PCl_5 on 16:17-dihydroxyviolanthrone (I: X = Y = OH; A = B = H) in boiling *o*-dichlorobenzene leads to the formation of a number of new vat dyes reputedly having the structures given below—(*a*) (II: A = Cl, B = H) and (*b*) (II: A = B = Cl) are obtained in admixture and dye cotton blue from a blue–violet vat; (*c*) (I: X = OH, Y = Cl; A = B = H) and (*d*) (I: X = OH, Y = A = Cl, B = H) in admixture dye cotton greenish grey–blue from a blue, weakly fluorescent vat. (*e*) (I: X = Y = Cl, A = B = H) dyes cotton violet–blue from a blue, weakly fluorescent vat. (*f*) (I: X = Y = A = Cl, B = H) dyes cotton greyish blue from a blue vat.



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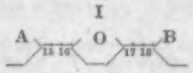


II

A. J.

4-Chloropicolinic Acid. H. S. Mosher and M. Look. *J. Org. Chem.*, **20**, 283–286 (March 1955).

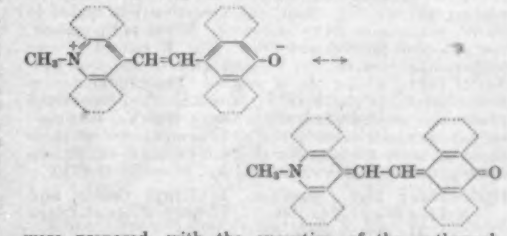
Picolinic acid hydrochloride on treatment with thionyl chloride in presence of SO_2 leads to the 4-chloro deriv., several hydrazides of which are prepared. H. E. N.

Reactivity of Polynuclear Aromatic Amines. F. L. Sixma. *Rec. Trav. chim.*, **74**, 168–174 (March 1955).

Log *k* for the reaction of 2 : 4-dinitrochlorobenzene with aniline, 1- and 2-naphthylamines, 1-, 2-, and 9-aminoanthracenes, and 1- and 9-aminophenanthrenes is inversely proportional to the localisation energy which may be calculated by the L.C.A.O. molecular orbital method, or to the charge density at the extra cyclic atom in the hydrocarbon anologues of the amines. In the case of 4-aminophenanthrene, however, the relationships do not hold, because of the steric hindrance. C. H. R.

Colour and Constitution. II—Phenolbetaines. S. Hünig and O. Rosenthal. *Liebigs Ann.*, **592**, 161–179 (April 1955).

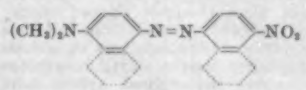
The nine dyes represented by the formula—

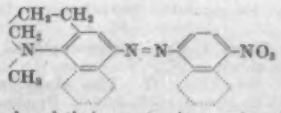


were prepared, with the exception of the anthranol–acridine cpd., and their spectra in a series of solvents of different dielectric constant determined. The three cpd. of lower mol. wt. exhibit a negative solvatochromic effect (i.e. an increase in the dielectric constant of the medium has a hypsochromic effect), the others a positive one. The results are discussed.

III—Polarisable Azo Dyes. S. Hünig and K. Requardt. *Ibid.*, 180–189.

The eight dyes, four represented by each of the formulae—





were prepared and their spectra in a series of solvents of different dielectric constant determined. All cpd. exhibit a positive solvatochromic effect, indicating that the structure of these dyes is very near to that of the classical formulae, but minor variations are noted. Steric hindrance to the coplanarity of the dialkylamino group, but not the nitro group, is evident. H. E. N.

Decomposition of Mixed Diazonium Fluoroborates. J. C. Brunton and H. Suschitzky. *J.C.S.*, 1035 (March 1955).

Diazonium fluoroborates in admixture appear to decompose without appreciable interaction. *p*-Carboxy-admixed with *o*-nitro-benzenediazonium fluoroborate affords a 40% yield of *p*-fluorobenzoic acid. The mixed decomposition method is found convenient for the simultaneous preparation of two separable aromatic F compounds. A mildly decomposing fluoroborate appears to be an ideal diluent for the decomp. of diazonium fluoroborates containing the NO_2 group, since it transforms the otherwise violent pyrolysis into a smooth reaction.

H. H. H.

Reaction of Diazo Compounds with Sulphamic Acid and its Derivatives. I—Reaction of Diazo Compounds with Methylsulphamic Acid. II—Properties of 1-Aryl-3-methyl-3-triazenesulphonic Acids. D. Z. Zavel'skiĭ and L. A. Lishnevskaya. *Sbornik statei obshchei khim.*, 1, 434–445, 446–452 (1953): *Chem. Abs.*, 49, 905 (25 Jan. 1955).

Potassium methylsulphamate (preparation described) couples with diazonium salts at pH 6 to give crystalline products $Ar{\cdot}N{:}N{\cdot}N(CH_3){\cdot}SO_3K$. The reverse reaction, with regeneration of the diazonium salt, occurs in more strongly acid soln., the cleavage being slower when Ar contains negative groups. The cleavage can be used as a basis for the analysis of the triazenesulphonates.

A. E. S.

Sulphonated Triphenylmethane Dyes. S. Bodforss. *Kgl. Fysiograf. Sällskap. Lund, Förh.*, 24, (4), 1–6 (1954): *Chem. Abs.*, 49, 4993 (10 April 1955).

It is difficult to crystallise the pure Na, Ba, $Co(NH_3)_6$ and organic amine salts of sulphonated triphenylmethane dyes. The thiocyanate and cyanide salts are not known to exist. The K salts of Fast Green SF (I), Fast Green carbinol (II), New Acid Green 3BX (III) and Brilliant Acid Green 6B (FBy) (IV) were prepared by the methods used by Ciba and FBy. The K salts of I, III and IV were prepared by dissolving the Na salt in warm CH_3OH, cooling and filtering. Satd. aq. K acetate was added in 10-ml. portions at 24 hr. intervals. When pptn. ceased, the ppt. was filtered and dried. The K salt of II was prepared by treating the CH_3OH soln. of the dye with NaOH before adding the K acetate. The products were tested for C, H, N, S, K, O and Cl. They contained practically no inorganic Cl, whereas the Na salt may contain as much as 30% NaCl. It is claimed that these compounds are not complex salts but probably carbonium hydroxides or imonium hydroxides.

C. O. C.

Hydrophilic Dye Cyanides. I—Ethyl Green and Xylene Blue Cyanides. L. Chalkley. *J. Amer. Chem. Soc.*, 77, 1848–1850 (5 April 1955).

Addition of a hydrophilic group (quaternary ammonium or sulphonic acid) to an aminotriphenylmethane dye cyanide produces substances that are photosensitive in aqueous solution. Ethyl Green (*C.I.*685) cyanide was prepared by conventional reaction of the dye with NaCN at room temperature. Xylene Blue VS and AS (*C.I.*672 and 673) did not react readily with NaCN at room temperature but a good yield was obtained by heating under pressure. An aqueous solution of Ethyl Green cyanide on exposure to ultraviolet radiation behaves much like an alcoholic solution of Malachite Green cyanide. Aqueous solutions of the Xylene Blue cyanides showed more complex reactions than those shown by alcoholic solutions of basic dye cyanides. Colour formation was slower and, under suitable conditions, at least two dyes were formed from a single dye cyanide—the original Xylene Blue and in addition a dye having a different absorption band.

C. O. C.

Polymethin Dyes. I—The Cyanomethins. M. Strell, W. B. Braunbruck, W. F. Fühler, and O. Huber. *Ann.*, 587, 177–194 (1954); **II—The Malocyanines.** M. Strell, W. B. Braunbruck, and L. Reithmayr. *Ibid.*, 195–206: *Chem. Abs.*, 49, 4645–4648 (10 April 1955).

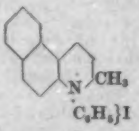
I—An account of successes and failures in synthesising many cyanomethin dyes. The structure and nomenclature of the various dyes are discussed and their absorption spectra given. With increase in the number of CH:CH groups, λ_{max} shifted towards longer wavelengths, each additional CH:CH causing a change of 100–110 mμ.

II—The malocyanines are neutral colouring matters and form no salts. They have in their structure the $(>CHCH<)_nCR(CN)$ group linked to a heterocycle (n = 1–3, seldom 4; R = CN or $COOC_2H_5$). They are dipolar halochromic dyes. Their structure is compared with that of the cyanines, merocyanines and sym. cyanomethins. The synthesis of 8 such dyes is described.

C. O. C.

Synthesis of 3-Methyl-4-phenylbenzo[*f*]quinolinium Iodide, and some of its Reactions. G. T. Pilyugin and E. P. Opanasenko. *Ukrain. khim. zhur.*, 18, 625–630 (1952): *Chem. Abs.*, 49, 1043 (25 Jan. 1955).

Synthesis of 3-methyl-4-phenylbenzo[*f*]quinolinium iodide (I) is based on the reaction of *N*-phenyl-2-naphthylamine with paraldehyde. By the application to I of well known reactions for the conversion of quinaldinium deriv. into cyanine and related dyes, the following products are obtained— bis[4-phenyl-3-benzo[*f*]quinoline]trimethincyanine iodide (λ_{max} 645 mμ. in ethanol); [4-phenyl-3-benzo[*f*]quinoline][1 : 3 : 3-trimethyl-2-indolenine]trimethincyanine perchlorate (λ_{max} 580 mμ.); 4-[phenyl-3-benzo[*f*]quinoline][3-ethyl-2-benzothiazole]trimethincyanine iodide (λ_{max} 590 mμ.); [4-phenyl-3-benzo[*f*]quinoline][3-ethyl-2-benzothiazole]monomethincyanine iodide (λ_{max} 506 mμ.); 3-(*p*-dimethylaminostyryl)-4-phenylbenzo[*f*]quinolinium iodide (λ_{max} 522 mμ.). These *N*-C_6H_5 cpd. are more deeply coloured than the corresponding *N*-C_2H_5 cpd.

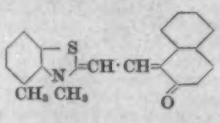


(I)

A. E. S.

Effect of the Solvent on the Colour of Dyes—III. A. I. Kiprianov and E. S. Timoshenko. *Ukrain. khim. zhur.*, 18, 347–357 (1952): *Chem. Abs.*, 49, 984 (25 Jan. 1955).

Seventeen new dimethin dyes are prepared, and the absorption spectra of their soln. in various solvents are determined. The variations observed are discussed in their relation to changes in the polarity of the solvent and the effect of these changes on the polarisation of the dye mol. One of the dyes examined is of formula—

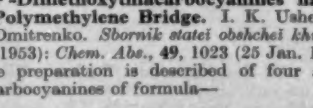


and is prepared by refluxing 2:3:4-trimethylbenzothiazolium methosulphate with 2-hydroxy-1-naphthaldehyde in ethanol in presence of a little piperidine. It gives pink soln. in water, reddish-violet soln. in ethanol (λ_{max} 579 mμ.), and violet soln. in chloroform (λ_{max} 590 mμ.). The other dyes, which are analogous in structure and behaviour, are prepared by suitable variations of this method. Derivatives of naphtho[2,1]thiazole and naphtho[1,2]thiazole and of 1:4-naphthaquinone, *o*-benzoquinone, and *p*-benzoquinone are included.

A. E. S.

***ar*:*ar*′-Dimethoxythiacarbocyanines having an αγ-Polymethylene Bridge.** I. K. Ushenko and I. P. Dmitrenko. *Sbornik statei obshchei khim.*, 1, 650–656 (1953): *Chem. Abs.*, 49, 1023 (25 Jan. 1955).

The preparation is described of four *xx*′-dimethoxythiacarbocyanines of formula—

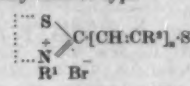


(X = anion). When n = 2 and x = 4, 5, and 6, λ_{max} = 604, 615, and 618 mμ. respectively; when n = 3 and x = 4, λ_{max} = 588 mμ.

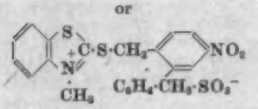
A. E. S.

Compounds containing Sulphur Chromophores. I—Action of Bases on Heterocyclic Sulphide Quaternary Salts. E. B. Knott. *J.C.S.*, 916–927 (March 1955).

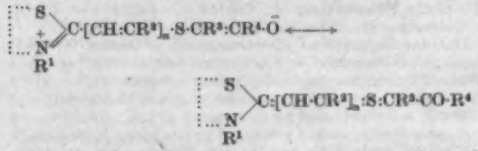
Quaternary salts of type—



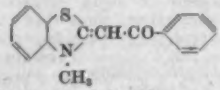
or



when treated with bases do not give the sulphides—



as required for compounds containing chromophoric S. The reaction eliminates S with formation of amide-type resonators, e.g.—

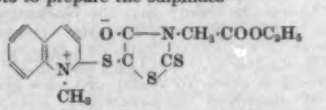


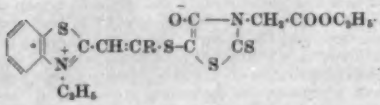
A mechanism is proposed based on an intramolecular condensation with intermediate episulphide formation.

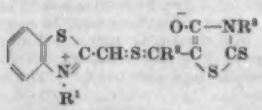
H. H. H.

II—Attempts to prepare Sulphide Analogues of *mero*Cyanines. E. B. Knott and R. A. Jeffreys. *J.C.S.*, 927–933 (March 1955).

Attempts to prepare the sulphides—







analogous to *mero*cyanines but in which a S atom is inserted in the bridge between the two end nuclei, have given only the *mero*cyanines themselves. This elimination of the S atom is analogous to the reaction described in I (preceding abstract). In some cases *mero*cyanine disulphides are isolated.

H. H. H.

III—Experiments on the Synthesis of Ionic Dyes. E. B. Knott. *J.C.S.*, 933–937 (March 1955).

Attempts to obtain cationic and anionic sulphides, designed to show whether the S atom is capable of functioning as a chromophore, have led only to the parent cyanines and oxonols.

H. H. H.

IV—Derivatives of Heterocyclic Methylene Bases. E. B. Knott. *J.C.S.*, 937–949 (March 1955).

The synthesis of a series of substituted 2-methylene deriv. of 3-methylbenzothiazoline and of 1:2-dihydro-1-methylquinoline is described. Alcoholic triethylamine readily removes S from the 2-benzoylthiomethylene deriv. to give the 2-phenacylidene analogues. The stabilities of such sulphides, in which a $+M$ and a $-M$ group are linked by an S atom, are compared with those of related compounds and are correlated with structures. The absorption spectra of these compounds indicate that an S atom may utilise its *d*-orbitals and function as a resonance transmitter, although it is not equiv. to a vinylene group. An O atom may behave similarly.

H. H. H.

V—Complex Cyanines. E. B. Knott. *J.C.S.*, 949–954 (March 1955).

The preparation of complex cyanines derived from 1:3-dithiacyclopentan-4-one is described. Their absorption spectra indicate that the thiol-ester (·CO·S·) bridge of the above nucleus functions as an efficient transmitter of electrons between the auxochromes of the dyes, without utilising the *d*-orbitals of the S atom. These absorptions are compared with those of related complex cyanines derived from tetrahydroglyoxaline and thiazolid-4- and -5-one.

H. H. H.

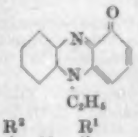
Formation of a Phenazine Ring. Yu. S. Rozum. *Ukrain. khim. zhur.*, **16**, (4), 434–440 (1950): *Chem. Abs.*, **49**, 1063 (25 Jan. 1955).

2:2′-Dinitrodiphenylamine heated in methanol with Na yields 5:10-dihydrophenazine, reduced by Zn to phenazine. Various substituted phenazines are prepared analogously.

A. E. S.

Absorption Spectra and Structure of *N*-Alkylphenazinones. Yu. S. Rozum. *Sbornik statei obschei khim.*, **1**, 600–606 (1953): *Chem. Abs.*, **49**, 1065 (25 Jan. 1955).

Photochemical oxidation of 1-ethoxy-5-ethylphenazine ethosulphate (I) yields a mixture of II (blue) and III ($R^1 = R^2 = H$) (red). Oxidation of I with ferricyanide yields a mixture of III ($R^1 = O{\cdot}C_2H_5$, $R^2 = H$) and III ($R^1 = H$, $R^2 = O{\cdot}C_2H_5$) (both red). Products of the oxidation of various dialkoxyphenazine salts are investigated and identified.



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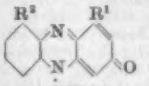


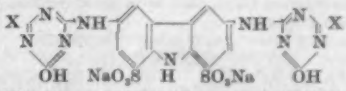
(III)

A. E. S.

Syntheses of Organic Fluorescent Compounds. XXI—Synthesis of Cyanuric Chloride. Z. Yoshida and R. Oda. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **59**, 92–94 (1953); **XXII—Syntheses of *s*-Triazine Derivatives of 3:6-Diaminocarbazoledisulphonic Acid.** Z. Yoshida, Y. Kato, and R. Oda. *Ibid.*, 161–162: *Chem. Abs.*, **49**, 4679 (10 April 1955).

XXI—Cyanuric chloride is commonly used to introduce the triazine ring in organic fluorescent compounds to increase the fastness to light. Of four methods of making cyanuric chloride investigated that from cyanuric acid and PCl_5 is best, the yield being 81·6 and 57·1% based on the acid and urea respectively, the cyanuric acid being prepared from urea and crude $ZnCl_2$.

XXII—Six *s*-triazine derivatives of 3:6-diaminocarbazoledisulphonic acid—



(X = NHC_6H_5, $NHC_6H_4CH_3$-*o*, $NHC_6H_4SO_3Na$-*p*, NHC_6H_4COONa-*o*, $N(CH_3)_2$ and $N(C_2H_5)_2$). They all showed bluish violet fluorescences of medium to weak intensity, inferior to those of the corresponding derivatives of 4:4′-diamino-2:2′-disulphonic acid.

C. O. C.

9 : 10-Disodio-9 : 10-dihydroanthracene. C. S. Rondestvedt and I. Nicholson. *J. Org. Chem.*, **20**, 346–351 (March 1955).

The compound is prepared using a dispersion of sodium in tetrahydrofuran and on carbonation affords *cis*-9:10-dihydroanthracene-9:10-dicarboxylic acid in 70% yield. Otherwise the disodio adduct appears to have synthetic value only in its reactions with alkyl halides and possibly with aldehydes and ketones, but transmetallation and hydrogen abstraction are its preferred modes of behaviour.

H. E. N.

Dyes for Acetate Rayon. III—Syntheses of Unsymmetrically Substituted 1:4-Bis(2-hydroxyethylamino)anthraquinones. Y. Bansho. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **57**, 510–512: *Chem. Abs.*, **49**, 4994 (10 April 1955).

1-Methylamino-4-(2-hydroxyethylamino)anthraquinone (25·2 g.) was obtained by heating leucoquinizarin (24·2 g.), 30% CH_3OH (15·1), monoethanolamine (6·72) and butanol (190) at 95°c. for 8 hr. and then steam distilling and purifying with 20% HCl. It is a blue dye which spectrophotometric comparison showed to be similar to Cellutate Brilliant Blue B. leucoQuinizarin (24·2 g.), water (120 c.c.), monoethanolamine (16·3 g.) and *p*-phenylenediamine (32·7) heated at 100°c. for 12 hr., dissolving in nitrophenol, treating with $SOCl_2$ (0·5 cc.) at 150°c. for 30 min. and purifying with 7% HCl yielded 1-(2-hydroxyethylamino)-4-(*p*-aminoanilino)anthraquinone (24·3 g.) a blue–green dye for acetate rayon. C. O. C.

Anthraquinone-acridone. II—Halogen Derivatives. K. Murata and K. Harada. *Bull. Fac. Engr., Hiroshima Univ.*, **3**, 131–134 (1954): *Chem. Abs.*, **49**, 5476 (25 April 1955).

Syntheses of various vat dyes derived from 2:1-anthraquinoneacridone are described. With the monobromo derivatives the general tendency is for the colour to become deeper the further bromination occurs from the NH group. The 3′:4′- and 4′:6′-dibromo derivatives (pink red) have a lighter colour than the 3′:6′- and 4′:5′-dibromo (brown and purple red respectively) derivatives. This change in colour was not observed with the 3′:4′:5′-, 3′:4′:6′- and 3′:5′:6′-tribromo derivatives which are all pinks. The 3′:4′:5′:6′-tetrabromo derivative has especially deep colour (dark brown). C. O. C.

Cyclisation of 3-Aroyl*meso*benzanthrones to 4:5-9:10-Dibenzopyrene-3:8-quinone and its Derivatives. A. J. Backhouse and W. Bradley. *J.C.S.*, 849–852 (March 1955).

The formation of a quinonoid diketone from 3-α-naphthoyl*meso*benzanthrone and KOH is confirmed under different conditions from those reported in *GP* 446,187. The action of KOH on 3-benzoyl-, 3-*m*-nitrobenzoyl-, and 3-α-naphthoyl-*meso*benzanthrone indicates the occurrence of three concurrent reactions, viz. (*a*) hydrolysis of acyl groups to give *meso*benzanthrone and a carboxylic acid, (*b*) cyclisation of the ketones to 4:5-9:10-dibenzopyrenequinone or a deriv. thereof, and (*c*) nuclear hydroxylation. H. H. H.

Purpurogallin. XII—Action of Organometallic Compounds on the Tropolones. R. D. Haworth and P. B. Tinker. *J.C.S.*, 911–915 (March 1955).

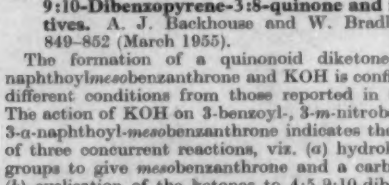
Kinetics of the Formation of Azobilirubin. J. T. G. Overbeek, C. L. J. Vink, and H. Deenstra. *Rec. Trav. chim.*, **74**, 85–97 (Feb. 1955).

Bilirubin (I) couples with diazotised sulphanilic acid to give azobilirubin (II) by the following sequence of reactions, both of which are of second order—

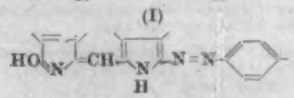
Bilirubin + diazo → azobilirubin + hydroxypyrromethenecarbinol (III) ($k = 8{\cdot}1 \times 10^3$ mole^{-1} min.$^{-1}$)

Hydroxypyrromethenecarbinol + diazo → azobilirubin + CH_2O ($k = 1{\cdot}2 \times 10^3$ mole^{-1} min.$^{-1}$)

A similar reaction takes place with 2:4:6-tribromobenzenediazonium sulphate.



(I)



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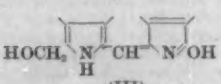


(III)

C. H. R.

Colouring Matters of the Aphididae. XII—Addition Reactions of Erythroaphin-*sl* and its Conversion into Erythroaphin-*fb*. B. R. Brown, A. Calderbank, A. W. Johnson, S. F. MacDonald, J. R. Quayle, and A. R. Todd. *J.C.S.*, 954–958 (March 1955).

XIII—Structure of the Erythroaphins. B. R. Brown, A. Calderbank, A. W. Johnson, B. S. Joshi, J. R. Quayle, and A. R. Todd. *J.C.S.*, 959–965 (March 1955).

All available evidence indicates that two structural formulae are possible for erythroaphin which differ in the location of an angular methyl group. These structures contain two dioxan rings fused to a tetrahydrocoronene nucleus, and this is supported by infrared spectroscopic evidence. H. H. H.

Treated Rutile-type Titanium Dioxide Pigments and their Properties. J. Taylor. *J. Oil & Col. Chem. Assocn.*, **38**, 233–249 (May 1955).

The development of aftertreatment processes for TiO_2 pigments is discussed and a typical flow-sheet diagram is given. The improvement in pigment properties effected by the removal of oversize particles, surface treatment, and washing, are illustrated by fineness-of-grind, viscosity, and flow measurements on pastes and paints made in ball mills, pug mills, and triple-roll mills. The improved results obtained with pigments of lower water-solubles content are illustrated by water dispersion tests and assessment of the blister resistance of metal primers. Natural and accelerated exposure results show that the resistance to chalking of rutile-type pigments is improved by the after-treatment process. An accelerated chalking test method is described. Aftertreatment improves covering power and, therefore, the cost of opacity. J. W. D.

Light Fastness of Lead Chrome Pigments. V. Watson and H. F. Clay. *J. Oil & Col. Chem. Assocn.*, **38**, 167–177 (April 1955).

Work is described in which lead chromes of various types were irradiated with a mercury-vapour lamp, the evolved gases being collected and analysed. The results obtained confirm the theory that the blackening of lead chromes is due to photoreduction; the oxygen evolved appears to be in the atomic form, and not molecular as suggested by Lashof (*J. Chem. Physics*, **11**, 196 (1943)). Brief reference is made to recent processes giving lead chromes of improved light-stability, and tentative suggestions are made regarding the mode of action of the treating agents used. The crystallography of lead chromes is summarised, and the theory that the blackening of lead chromes involves a change of crystal structure is refuted. Chromes having similar chemical compositions but different degrees of resistance to darkening reveal no differences when examined by X-ray methods; minor, and at present undetectable, lattice differences may be responsible. The X-ray pattern of a chrome is unchanged after "treating", and this suggests that the treating material is present simply as a coating, and does not enter the lead chromate lattice. J. W. D.

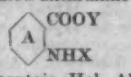
PATENTS

3-Hydroxy-2-naphtho-β-naphthylamide. ICI. *BP* 728,758

In preparing the β-naphthylamide of 3-hydroxy-2-naphthoic acid by condensing the latter with Tobias acid in an inert solvent with PCl_3 (*BP* 700,024; J.S.D.C., **70**, 95 (1954)) a good yield is obtained by carrying out the reaction in presence of Na_2HPO_4. E. S.

Diazoamino Compounds for Production of Azoic Dyeings and Prints by Neutral Steaming. Fran. *BP* 729,156

The diazoamino compounds prepared by condensing diazotised amines more strongly basic than 2:5-dichloraniline with *N*-substituted anthranilic acid derivatives—



(the nucleus A may contain Hal, Alk or OAlk groups; Y = H or an alkali metal; X = subst. or unsubst. Alk or *cyclo*alk, e.g. CH_2 or $CH_2{\cdot}COOY$), couple on cellulose fibres with suitable coupling components by steaming under neutral conditions. Thus the diazoamino compound derived from diazotised 5-chloro-2-aminotoluene and *N*-

o-carboxyphenylglycine is mixed with 3-hydroxy-2-naphtho-4′-methoxy-2′-methylanilide and Na_2SO_4, and the mixture dissolved in aq. NaOH and suitably thickened. The paste so obtained is printed on cotton, and the red azoic dye developed by steaming in the Mather–Platt for 6–7 minutes. A similar result is obtained by simply leaving at room temperature for 24 hr. or steaming for 30 sec. with acetic or formic acid vapour.

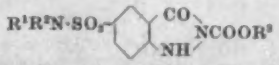
BP 729,157
BP 729,158

Preparation of the diazoamino compounds described in *BP* 729,156 (preceding abstract) by combining a suitable diazotised amine with an appropriate *N*-substituted anthranilic acid, and their isolation, are described. Thus diazotised 5-chloro-2-aminotoluene is added slowly at −2°C. to −3°C. to a soln. of the mono-sodium salt of *N*-*o*-carboxyphenylglycine in aq. Na_2CO_3. The mixture is then neutralised gradually with aq. NaOH. After filtering off some insoluble matter, the diazoamino compound is pptd. by addition of NaOH. This method of pptn. is of general application to the diazoamino compounds derived from derivatives of *N*-phenylglycine, which cannot be isolated by salting out or by evaporation *in vacuo*.

E. S.

Colour Couplers. ICI. *BP* 729,505

Compounds of formula—



(R^1 and R^2 = H or hydrocarbon; R^3 = hydrocarbon; when R^1 = H and R^2 = 1-naphthyl the latter must be substituted in the 4-position), e.g. 2-carbethoxy-5-(*N*-*p*-dodecylphenylsulphamyl)-indazolone, are useful photographic colour couplers as because of the pronounced bathochromic effect of the sulphonamide group they yield magentas when developed with aromatic amino developers, e.g. *p*-diethylamino aniline.

C. O. C.

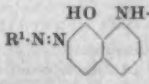
Chromable Blue–Green Monoazo Wool Dyes. Ciba. *BP* 728,236

Monoazo dyes 4-acylamino-2-amino-6-nitrophenol → 2:8-dihydroxynaphthalene-6-sulphonic acid may be chromed in substance, in the dyebath or on the fibre, to give blue–greens on wool, nylon, etc. Depending on the nature and quantity of the alkali used in coupling the products vary in some respects because coupling may occur at either the 1- or the 7-position. The acyl group may be aliphatic or aromatic, but is preferably the acetyl group. Thus diazotised 4-acetylamino-2-amino-6-nitrophenol coupled in presence of sodium carbonate gives a blue–green afterchrome dye for wool.

E. S.

Metallisable Monoazo Dye for Wool, Nylon, etc. S. *BP* 728,259

Monoazo dyes which may be metallised with Cr or Co compounds are made by coupling diazotised *o*-amino-hydroxy compounds of the benzene or naphthalene series with suitable *N*-acyl derivatives of 1-amino-8-naphthol under strongly alkaline conditions, so that coupling takes place in the 7-position. The products—

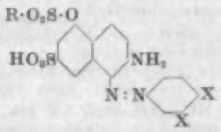


(R^1 = benzene or naphthalene series radical with an OH group *ortho* to the azo link; R^2 = H or lower alkyl, including halogen, alkoxy, aryloxy or carboxy derivatives of $CH_2.C_2H_4.C_2H_7$ or C_4H_9; or R^2CO = fumaryl), dye wool, nylon, etc. blue, green, or grey by the afterchrome or metachrome process, and may also be converted in substance into Cr or Co complexes. In alternative methods of preparation, monoazo dyes R^1NH_2 → 1-amino-8-naphthol may be *N*-acylated, or R^1NH_2 → 1:8-naphthalenediamine is boiled with dil. acid to convert the 8-NH_2 group into OH and the product is then *N*-acylated. Coupling in all cases is carried out under strongly alkaline conditions to ensure introduction of the azo group in the 7-position, as coupling in the 5-position gives browns of poor light fastness. Thus, all parts being by weight, 1-acetylamino-8-naphthol (5) is dissolved in 23% aq. NaOH (50) at 5°C., and 42% aq. NaOH (57) added, followed by 1-diazo-2-naphthol-4-sulphonic acid (5·8). Coupling is accelerated by adding small quantities of water, and the dye isolated by adding water (500) followed by dil. HCl. It dyes animal fibres blue from an acid bath, converted to greenish-grey by afterchroming.

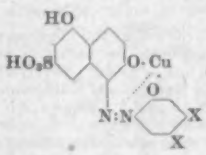
E. S.

Metal(Copper)-complex Monoazo Dyes and Intermediates. S. *BP* 727,747

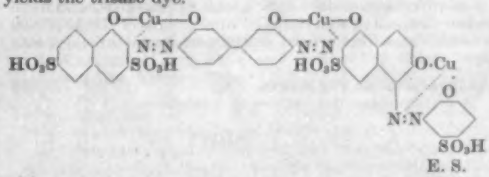
Monoazo compounds—



(one X = H, Hal, Alk, OAlk, or NO_2; the other X = SO_3H) obtained by acid-coupling *O*-arylsulphonyl esters of J acid with suitable diazo compounds are converted into copper complexes—



by replacing the NH_2 group by OH by treating with nitrous acid, coppering in presence of an oxidising agent, and finally hydrolysing the R·O_2S·O group. These copper complexes couple with suitable diazo compounds to give dis- and poly-azo dyes. Thus the monoazo compound metanilic acid → *O*-*p*-toluenesulphonyl-J acid (acid coupled) is treated with cold nitrous acid overnight and then converted into a copper complex by treatment cold with copper sulphate, copper acetate, and hydrogen peroxide. Hydrolysis with dil. aq. NaOH at 85°C. splits off the *O*-acyl group. The monoazo copper complex so formed is coupled with the diazoazo compound obtained by coupling 1 mol. of tetrazotised dianisidine with 1 mol. of 1-naphthol-3:6-disulphonic acid. Coppering under conditions which demethylate the OCH_3 groups of the dianisidine residue then yields the trisazo dye.



E. S.

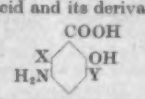
Metallised Pyrazolone Monoazo Dyes containing a Sulphonamido Group—Neutral Milling-fast Dyes for Wool, Nylon, etc. S. *BP* 727,528

A 1-amino-2-hydroxybenzene containing a SO_2NHR (R = H, Alk or phenyl) in the 3-, 4- or 5-positions is diazotised and coupled with a 1-phenyl-3-methyl-5-pyrazolone. The resulting monoazo dye is treated with a compound which yields Cr or Co to produce yellow to red neutral-dyeing dyes for wool and nylon, fast to milling and light. Thus 1-amino-2-hydroxy-3-*N*-methylsulphonamide is diazotised and coupled with 1-(2′:5′-dichlorophenyl)-3-methyl-5-pyrazolone in presence of aq. Na_2CO_3. The precipitated dye is filtered off, refluxed for several hr. with aq. $Na_2Cr_2O_7$ in presence of NaOH and finally separated by adding salt.

R. K. F.

Chromable Disazo Dyes for Wool. FH. *BP* 729,740

5-Aminosalicylic acid and its derivatives—

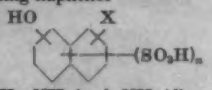


(X and Y = H, CH_3, or Cl) are diazotised and coupled with suitable middle components and the aminomonoazo compounds so formed diazotised and coupled with

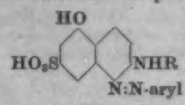
coupling components to give disazo dyes which may be metallised, particularly with chromium compounds, in substance, on the fibre, or in the dyebath. Thus the disazo dye 5-aminosalicylic acid → α-naphthylamine → N.W. acid dyes wool claret, converted to reddish navy by after-chroming. E. S.

Blue and Navy Blue Metal(Copper)-complex Trisazo Direct Cotton Dyes. S. *BP* 727,745

The copper complexes of trisazo dyes obtained by coupling tetrazotised 3:3′-dialkoxybenzidines with 1 mol. of an *ortho*-coupling naphthol



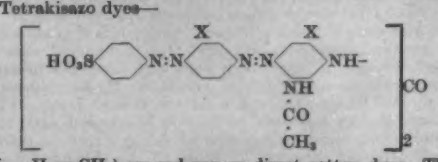
(X = H, OH, NH_2, NH Acyl, NH Alk, or NH Aryl; *n* = 1 or 2) and 1 mol. of a monoazo compound



(R = H, Alk, or a radical of the benzene series; aryl is of the benzene or naphthalene series in which the *ortho* position to the azo link is free of COOH) dye cellulose blue or navy blue. Thus tetrazotised dianisidine is coupled first with 1 mol. of 1-naphthol-3:6-disulphonic acid, and then with 1 mol. of 2-amino-1-phenylazo-5-naphthol-7-sulphonic acid in presence of soda ash and pyridine. Boiling with aq. cuprammonium hydroxide then yields a navy blue direct cotton dye. E. S.

Orange Direct Cotton Tetrakisazo Dyes. FBy. *BP* 727,421

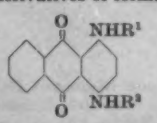
Tetrakisazo dyes—



(X = H or CH_3) are red-orange direct cotton dyes. They are prepared by condensing 2 mol. of an appropriate disazo dye, e.g. 4-aminoazobenzene-3:4′-disulphonic acid → *m*-aminoacetanilide, with 1 mol. of phosgene, and have better fastness to acid than the similar dyes of *BP* 13727/09 in which cresidine is used as end component in the disazo dye. E. S.

Anthraquinone Pigments. FBy. *BP* 727,996

Anthraquinone derivatives of formula—



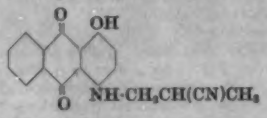
(R^1 and R^2 = organic radicals containing together ≮ 2 NH_2 groups; or R^1 or R^2— not both— may be H), formed by condensing quinizarin, leucoquinizarin or leuco-1:4-diaminoanthraquinone with a polyamine, are condensed with a polyfunctional compound, e.g. a di-isocyanate, capable of reacting with NH_2 groups. The products are violet, blue and green pigments insoluble in organic solvents. Thus, 1:4-bis-aminoethylaminoanthraquinone (from leuco-1:4-diaminoanthraquinone and ethylene diamine) is heated at 100–150°c. in nitrobenzene with diphenyl carbonate. The resulting blue polyurea is filtered off and washed with methanol. R. K. F.

Leuco-Indanthrone Sulphuric Esters. American Cyanamid Co. *USP* 2,659,727

β-Aminoanthraquinone leuco sulphuric esters having a free α-position adjacent to the β-NH_2 are treated with a simple compound of Fe of valency 4 or 6, or of Co of valency 3 or 4, or of Ni of valency 3 or 4; to give leuco-indanthrone tetra-sulphuric esters. Thus, to an aq. solution of 2-amino-3-chloroanthraquinone-9:10-disulphuric ester, obtained by hydrolysis of the corresponding acetyl derivative, is added block cobaltic hydroxide, prepared by adding cobaltous acetate to a solution of KOH in 15% sodium hypochlorite and filtering. The mixture is heated to 85°c. till oxidation is complete, filtered and the tetra-sulphuric ester separated by adding KCl. R. K. F.

1-β-Cyanopropylamino-4-hydroxyanthraquinone— Disperse Dye. Eastman Kodak. *USP* 2,659,739

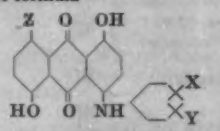
A mixture of 1:4-dihydroxyanthraquinone and its leuco derivative is heated at 120°c. in *n*-butanol or isopropanol under pressure with β-cyanopropylamine, made by condensing methacrylonitrile under pressure with aq. NH_3, to give the violet disperse dye—



fast to light and burnt gas fumes. R. K. F.

1:5-Dihydroxy-4-halogeno-8-hydroxyalkylphenyl-aminoanthraquinones— Disperse Dyes. Eastman Kodak. *USP* 2,659,740

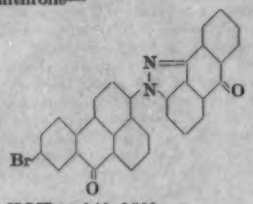
Compounds of formula—



(X = monohydroxyalkyl; Y = H, Cl, OH, CH_3O, or CH_3; Z = F, Cl or Br) are blue disperse dyes, fast to light and burnt gas fumes. They are made, for example, by heating 1:5-dichloro-4:8-dihydroxyanthraquinone with *p*-aminophenylethyl alcohol at 170°c. for 4 hr. R. K. F.

Benzanthronyl-Pyrazolanthrone Derivatives— Vat Dyes. American Cyanamid. *BP* 728,572

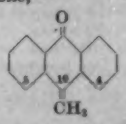
A navy blue vat dye faster to light than dibenzanthrone is obtained by fusing 8-bromo-(or chloro-)-3-benzanthronyl-Py-1-pyrazolanthrone—



with alcoholic KOH at 140–150°c. R. K. F.

Ethylene-bridged Dibenzanthronyls and derived Vat Dyes. Eastman Kodak. *USP* 2,659,735

10-Methyleneanthrone,



and derivatives which are unsubstituted at the 4 and/or 5 positions are condensed with 1:3-*cyclo*hexadiene in presence of a mild oxidising agent to give 2,2′-ethylene-bridged 3,3′-dibenzanthronyls, which on fusing with alkali and oxidising are converted into vat dyes. Thus 10-methylene-anthrone and 1:3-*cyclo*hexadiene are refluxed in nitro-benzene for 1 hr. The product, which is separated by adding ethanol, is heated to a final temperature of 140–145°c. for 2 hr. in a mixture of KOH and ethanol to give a blue vat dye. Alternatively, $NaNO_2$ is added to the melt when a dull red light-fast vat dye of constitution—

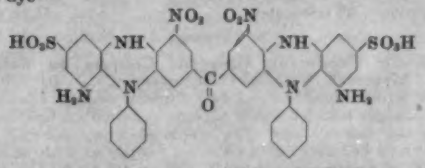
is obtained. R. K. F.

Halogenated Acedianthrone-Carbazoles—Vat Dyes. CFM. *BP* 728,554

The acedianthrone anthrimides of *BP* 410,552 and 453,853 are cyclised to the corresponding carbazoles and halogenated to produce brown vat dyes of good fastness to ironing and water-spotting. Thus the anthrimide obtained from αα′-dichloroacedianthrone and α-aminoanthraquinone is heated at 80–85°c. for 3 hr. in a melt of $AlCl_3$ and $NaHSO_3$ through which SO_2 has been passed. Bromine is next run in slowly after adding a small quantity of iodine, and the product separated by pouring on to ice. R. K. F.

Azine Wool Dyes. FH. *BP* 727,466

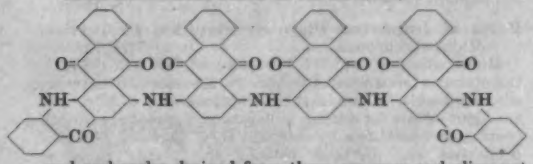
3:5:3′:5′-Tetranitrobenzophenone containing halogen or O Alk in the 4:4′-positions is condensed with 2 mol. proportions of a 2:6-diaminodiphenylamine-4-sulphonic acid and the product cyclised to an azine derivative which is a brown wool dye fast to light and wet treatments. Thus the dye —



is prepared by boiling 4:4′-dichloro-3:5:3′:5′-tetranitrobenzophenone and 2:6-diaminodiphenylamine-4-sulphonic acid in alcohol for several hr. in presence of sodium acetate. After cooling and filtering the resulting product is cyclised by heating at 90°c. in aq. Na_2CO_3. R. K. F.

Tetra-anthrimides containing two Acridone Rings and the corresponding Carbazoles—Vat Dyes. Ciba. *BP* 727,826

Tetra-anthrimides based on the structure—



and carbazoles derived from them are grey and olive vat dyes. They are made, e.g. by refluxing 4:4′-dibromo-1:1′-dianthrimide (from 4:4′-diamino-1:1′-dianthrimide by diazotising and Sandmeyer reaction), 4-amino-2:1(N)-1′:2′(N)-anthraquinonebenzacridone, sodium and copper acetates in nitrobenzene for 7 hr. The product is cyclised to the corresponding carbazole by heating at 160–165°c. in a mixture of $AlCl_3$ and NaCl. R. K. F.

Phthalocyanines—Modified Process of Making. General Aniline. *BP* 729,123

The yield and purity of phthalocyanine obtained in a process of the "urea-inert solvent" type are both improved by including in the reaction mixture < 25% by weight of the phthalocyanine intermediate (e.g. phthalic anhydride), of a monobasic aromatic sulphonic acid or one of its salts. Thus, all parts being by weight, phthalic anhydride (10·5), urea (12·5), ammonium molybdate (0·25), sodium xylene sulphonate (5·4) and $AlCl_3$ (3) are heated in trichlorobenzene (40) at 200–205°c. for 6 hr. Urea (5·4) and trichlorobenzene (10) are then added and heating continued for a further 5 hr. An 85% yield of chloroaluminium phthalocyanine is obtained.

BP 729,124

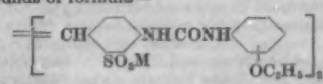
An aliphatic monobasic sulphonic acid containing 1–5 C, one of its salts, alcohol esters, amides or acid halides replaces the aromatic sulphonic acid in the preceding patent. R. K. F.

Chlorinated Copper Phthalocyanine. Ausbacher-Siegle Corpn. *USP* 2,662,085

Copper phthalocyanine is chlorinated by dissolving it in chlorosulphonic acid, and passing in Cl_2 in presence of a catalyst e.g. sulphur chlorides, I_2 or $FeCl_2$ below 115°c. Thus, a solution of copper phthalocyanine in chlorosulphonic acid containing S_2Cl_2 and ICl is maintained at 30°c. while passing in Cl_2. When the colour of the solution changes from olive to red the temperature is raised to 90°c. and passing of Cl_2 continued until a test sample separated by pouring into water has the desired greenish shade. R. K. F.

Fluorescent Brightening Agents. General Aniline. *USP* 2,676,982

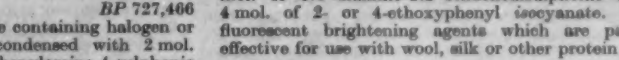
Compounds of formula—



(M = H or cation) are readily prepared by condensing 1 mol. of 4:4′-diamino-2:2′-stilbenedisulphonic acid with 4 mol. of 2- or 4-ethoxyphenyl isocyanate. They are fluorescent brightening agents which are particularly effective for use with wool, silk or other protein fibres. C. O. C.

Polymethin Dyes. Ilford. *BP* 728,078

Dyes of formula—



(R^1 and R^3 = Alk, aralkyl or Ar; R^2 = cyano or COR^4 (R^4 = alkoxy, aralkoxy, amino or subst. amino); x = 0 or 1; D^1 and D^2 = same or different, atoms to complete a nucleus of the benzthiazole, naphthathiazole, benzoxazole or indolenine series), e.g. 1:3-bis(3-methylbenzthiazolylidene) - 2 - (cyanocarbethoxymethylene) - propane, impart to silver halide emulsions strong sensitivity in the green and red regions of the spectrum. C. O. C.

Caramel Colour Composition. Union Starch & Refining Co. *BP* 729,219

A colouring composition having a tinctorial power of 30–50 is produced by the process of *BP* 698,105 (*J.S.D.C.*, **69**, 510 (1953)) and is then mixed with water and a stabiliser to produce a solution containing 2–20% stabiliser and 30–60% colouring compounds. The dimers and other polymers of propylene alcohol, the polyalkylene oxides by themselves or combined with sorbitan esters, e.g. the sorbitan monostearate polyoxyethylene derivative in which the polyoxyethylene portion comprises several CH_2CH_2·C-units, can be used as stabilisers, the preferable stabiliser being propylene glycol. C. O. C.

Water-dispersible Carbon Black. J. M. Huber Corpn. *BP* 729,381

Densified carbon black readily dispersible for use in colouring paper, water-based paints or inks, etc. is produced by mixing carbon black with > 1% by weight of bentonite. C. O. C.

Preventing Agglomeration of Particles of Natural Calcium Carbonate and rendering them more readily Dispersible. Société Anonyme du Blanc Omya. *BP* 728,698

Grinding the carbonate in presence of a fatty acid of > 11 C at a temperature not below that at which the two react results in quicker grinding and in a product of finer particle size which does not tend to agglomerate and which is readily dispersible. C. O. C.

Lead Chrome Pigments. Cromford Colour Co. *BP* 730,176

Lead chrome pigments of improved fastness to light are produced by treating lead chromate (with or without lead sulphate) with silicic acid or with silicic acid and/or aluminium hydroxide in either order. Probably the improvement in light fastness is caused by the silica or alumina in the pigment particles acting as a protective coating so as to retard photoreduction of the chromate to chromite. C. O. C.

Surface-coated Silicon-containing Particles as Reinforcing Pigments for Light-coloured or White Rubbery Compositions (XIII p. 431).

Colouring Vinyl Polymers Red (XIII p. 431).

V— PAINTS; ENAMELS; INKS

Sedimentation, Thixotropy, and Related Subjects—I and II. K. L. Wolf and R. Kurtz. *Farbe und Lack.*, **60**, 483–490, 528–530 (1954): *Chem. Abs.*, **49**, 4370 (10 April 1955).

I—Sedimentation studies were made by shaking 1·5 g. sifted pigment in 10 ml. of liquid, allowing it to settle and measuring the sedimentation when constant. Increasing particle size generally reduced the sedimentation vol., *S*,

of sieve-fractionated NaCl, KCl, kieserite and langbeinite (0·006–0·5 mm.) in CCl_4 or *cyclo*hexane but had little effect on it in acetone and methanol. Tables are given of data on behaviour of $MgCO_3$, $CaCO_3$, $SrCO_3$, $BaCO_3$, $SrSO_4$, $BaSO_4$, $Ba(NO_3)_2$, $PbCrO_4$, Cr_2O_3, Orange V, Paris Blue, Pigmosol Green, sugar, starch, cubic NaCl and KCl, octahedral NaCl (crystallised from a soln. containing urea), octahedral KCl (crystallised from a soln. containing $PbCl_2$), kieserite, schoenite, langbeinite, anatase and rutile in CCl_4, C_6H_6, C_6H_{12}, tetrahydrofuran and C_5H_{14} (i.e. non-polar liquids). NaCl, $SrSO_4$, $BaCO_3$ and $PbCrO_4$ settled rapidly as coarsely fluffy sediments of large S, whereas the reverse occurred in polar liquids, e.g. primary alcohols or amines, $HCONH_2$ in water. In liquids of intermediate polarity (methyl acetate, ether, and dioxan), S were fairly large to large and the structure was fluffy. In liquids with strongly polar functional groups (CH_3COCN, butyl cyanide and acetone), S were smaller and the structure was finely granular. The sediments were usually thixotropic in non-polar liquids but rarely so in polar liquids. The sedimentation rate varied considerably and independently of d. and viscosity of the medium. In non-polar liquids settling was often complete in fractions of a minute but in polar liquids settling took hours or days. Milky supernatant liquids often indicated a tendency to form stable suspensions. Some sediments, e.g. the alkali salts in C_6H_6, were transparent, some (in butylamine) were faintly coloured. Theory lead to the conclusion that S is only generally dependent on the dielectric constant of the liquid, but more so in presence of polar molecules having accessible polar groups. Even small additions of such liquids effect S in non-polar liquids. Thus, the sediment of Cr_2O_3 in CCl_4 (8 ml.) is changed from fluffy to fine by adding a few drops of octanol; the sedimentation rate and volume are noticeably decreased. Octylamine or butylamine have an even greater effect, the latter producing the behaviour found in butylamine itself. The polar liquid is thus sedimentation-active towards the solid in question. This activity is expressed as reduction of S, ΔS, by addition of 0·1 g. or 0·1 ml. of active material to 1·5 g. pigment in 10 ml. liquid, and in % of observed maximum reduction. The most effective sedimentation-active materials are octyl- and decylamines, stearic and oleic acids and octanol, ethyl hexanol, methanol, glycerol and butanediol. Some substances are sedimentation-active in water, e.g. $CO(NH_2)_2$, camphor, dioxan for $CaCO_3$ and $BaCO_3$, oxalic acid for Cr_2O_3; alcohols and acetamide are not sedimentation-active in water. Butylamine increased the S of $MgCO_3$ in water, and Hostapon (FH) reduced the sedimentation rate of all sediments, regardless of S. The S of rutile is increased by butylamine and slightly increased by oxalic acid, whereas S of anatase is unaffected by butylamine but sharply decreased by oxalic acid. Dependence of sedimentation-activity on concentration of active materials was studied for several systems— amines and acids were the most effective. The activity of some of these substances followed the Langmuir adsorption isotherm in accordance with theory. The adsorption constant K varied for 0·001 (schoenite/butylamine, schoenite/oleic acid, KCl/octylamine or KCl/oleic acid) to 0·1 ($CaCO_3$/octanol).

II— Effect of temp. (20–70°c.) on sedimentation of KCl, NaCl, schoenite and $CaCO_3$ in CCl_4, C_6H_6 and tetrahydrofuran varies considerably. In CCl_4 and in C_6H_6, S dropped with rising temp. for NaCl and KCl but no such simple function was found for the other solids (schoenite became dehydrated). In tetrahydrofuran the effect was generally small. Sedimentation rate and structure also varied. Sedimentation activity of butylamine in C_6H_6 for $CaCO_3$ or Cr_2O_3 dropped considerably with rise in temp. Heats of adsorption, calculated from charge in K, decreased. Association of the liquid molecules complicated sedimentation behaviour, e.g. in water or the lower alcohols. Thus minima of S were observed in brass pigment in soln. of butanol in *cyclo*hexane or of stearic acid in CCl_4. In the former system the minimum occurred at an extremely small mole fraction of butanol— similar to the effect of high-pressure lubricant additives and surface-active agents. Sedimentation activity of butylamine in CCl_4 was completely nullified by addition of water. This is probably caused by molecular combination between water and the amine; the effect was sensitive enough to be used to detect formation of complexes. In dioxan this effect was not seen, e.g. by adding water + acetamide. Thixotropy was not limited to leaflike solids, as is often thought, but was seen even with cubic and octahedral crystals in suitable liquids. Sedimentation is also related to electro-osmosis and both follow similar rules, thus the dielectric constant of a liquid is not the only factor (Coehn's rule) controlling it.

C. O. C.

High Polymer Films. V— Effects of Plasticisers, Resins, and Pigments on Viscoelastic Properties of Cellulose Nitrate Films. Y. Inoue. *Chem. High Polymers (Japan)*, **10**, 247–252 (1953): *Chem. Abs.*, **49**, 5020 (10 April 1955).

The Young's modulus of cellulose nitrate film increases with the pigment or resin content and decreases with the plasticiser content. Viscosity coefficients increase with addition of pigment. C. O. C.

Zinc Coating and Painting. W. Brachmann. *Metalloberfläche*, **8**, A157–160, A172–176 (1954): *Chem. Abs.*, **49**, 5855 (25 April 1955).

Review, 37 references. C. O. C.

PATENTS

Ink for Producing Different Colours on Record Material. National Cash Register Co. *BP* 729,242

A liquid vehicle carries two different organic adsorbate substances both of which become coloured on adsorption contact with one kind of inorganic adsorbent but only one of which becomes coloured when contacted with another kind of inorganic substance. Thus 3:3-bis-(*p*-dimethylaminophenyl)-phthalide and xanthene 9, *o*-benzoic acid, 3:6-bis-diethylamino-9-*p*-nitroanilinolactam dissolved in a mutual solvent become bluish green and red respectively on contact with attapulgite whereas calcium silicate produces a colour with only one of them. Thus by coating or impregnating parts of the second material with one or the other of these two inorganic substances, marks made with the fluid appear as purple or bluish green according to the impregnant present. C. O. C.

Paint of Improved Pigment Flotation Properties. Midland Silicones. *BP* 729,608

Modification of *BP* 706,719 (J.S.D.C., **70**, 325 (1954)). Copolymeric siloxanes obtained by the reaction between silicahydrosols, silane and/or siloxanes at $< 30°$c. are excellent agents for adding to paints or pigmented resinous coating compositions to decrease their pigment flotation properties and without causing the paints to show "cratering", i.e. formation of small bubbles, pockets or craters in the paint film as it dries. C. O. C.

Surface Coating Compositions. J. H. Pless. *BP* 728,957

Modification of *BP* 634,779. An aqueous solution containing ammonium sulphate and/or phosphate being used for the top coating composition instead of fluosilicate. This yields hard, water-insoluble, weather-resistant coatings on a wide variety of materials such as wood, stone, cement, etc. C. O. C.

Wrinkle Finish Coating Composition. New Wrinkle. *BP* 728,510

Wrinkle coating compositions which quickly dry at room temperature are obtained by cold blending a non-wrinkling varnish with raw tung oil. C. O. C.

Lacquers from Naphthalene-Formaldehyde Resins which yield Elastic Coatings. BASF. *BP* 730,248

Solutions containing (*a*) resins formed from naphthalene and HCHO and containing $< 5\%$ of O, and (*b*) difficultly-volatile, highly chlorinated paraffins are lacquers yielding elastic coatings. C. O. C.

VI— FIBRES; YARNS; FABRICS

Photodegradation of Cellulose, particularly in the Presence of Titanium Dioxide. E. Trieber. *Svensk Papperstidning*, **58**, 185–195 (31 March 1955).

A review of the present knowledge of the photolysis of cellulose and its photo-oxidation, with and without titanium dioxide present, is followed by an account of experimental work and a discussion of methods of reducing damage by light, and of determining the activity of the pigment. S. V. S.

Characteristics of Wool and Wool Fabrics. H. Bogarty, L. Fourt, and M. Harris. *Canadian Textile J.*, **72**, 63–66 (14 Jan. 1955).

Various typically wool-like characteristics, e.g. loft, thickness, surface hairiness, contact warmth, and resistance to wicking, are shown to depend on the random three-dimensional orientation of wool fibres in a fabric. This random arrangement occurs naturally with the use of wool by traditional methods and is aided by its unique properties, e.g. ability to felt, high elastic recovery, crimp, high fibre friction, and variability of size. It is considered that the production of more wool-like fabrics from blends and synthetic fibres may be achieved by methods which will promote more random structures, such as by use of crimp, heat-shrinking techniques, and modifications in fabric construction. W. K. R.

Differential Friction of Wool. P. Grosberg. *J. Textile Inst.*, **46**, T233–T246 (April 1955).

Theories advanced by Makinson and Mercer to explain the differential frictional effect (D.F.E.) of wool are critically examined, and a new hypothesis, based on the anisotropic elastic properties of moist keratin, is put forward. The deformation which takes place at the surface of an anisotropic scale depends on the direction of pulling, and, since a scale will be more readily compressed by a force at an angle than by one normal to the scale, a D.F.E. will result. The theory is expanded quantitatively, and an equation is developed which is shown to fit the experimental results of several workers. J. W. B.

Adsorption of Primary Alcohols by Wool. C. H. Nicholls and J. B. Speakman. *J. Textile Inst.*, **46**, T264–T269 (April 1955).

The adsorption isotherms on wool of methyl, ethyl, and *n*-propyl alcohols are determined, and are analysed by means of the Hailwood–Horrobin equation. It is found that the value of the molecular weight of the unit which combines with one molecule of adsorbate increases, and the fraction of the wool accessible decreases, with increasing size of the exploratory molecule, extreme values being 0·70 for water and 0·28 for *n*-propyl alcohol. Such data also provide further support for the Hailwood–Horrobin theory. J. W. B.

Felt from Man-made Fibres. H. G. Lauterbach. *Text. Research J.*, **25**, 143–149 (Feb. 1955).

A consideration of the properties of wool and fur fibres utilised in felt formation led to the conclusion that high elasticity and the ability to twist and entangle under a rubbing motion were the most important. Polyethylene fibres possess these properties to some extent, and it was found that a felt composed entirely of polyethylene fibre could be prepared by the conventional wool felt process. To obtain felts from other synthetic fibres, however, it was considered more profitable to alter the felting process to suit the fibre. In the modified felting process, fibre entanglement was achieved by needle-punching the carded batt instead of by the hardening operation in the conventional process, whilst fulling was replaced by heat shrinkage. Using Dacron fibres specially prepared to shrink 50–75% in length at 100°C. and following the process— carding, needle punching, heat shrinkage— a wide range of felts was produced. The properties of Dacron felts were controlled by the extent of fibre shrinkage, the amount of needle punching, and the weight of the carded batt. Dacron felts surpassed in strength and splitting resistance the specification for wool felts of similar weight and thickness. W. K. R.

Glass Fibres— Manufacture, Properties, and Uses. A. de Dani. *Chem. and Ind.*, 482–489 (30 April 1955).

The production of glass fibre in continuous filament and staple fibre form is described, accounts of the flame-drawing, centrifugal, and slag-wool processes being included. The tensile strength of short fibres is > that of long ones, owing to the smaller probability of the presence of flaws; strength is very largely independent of the diameter, but is influenced by the thermal history of the fibre. Existing evidence indicates that glass fibres are structureless, and consist of isotropic material. Glass fibres used in textile processing require protection against mechanical damage; a modern glass fibre size is composed of an oily substance for lubrication, a resin (usually thermoplastic) for bonding, and one or more cation-active agents to secure adhesion to the glass surface. In insulation products, dimensional stability is the main consideration, and binders in common use include starch, bitumen, and phenolic resins; photomicrographs are reproduced, showing glass fibres bonded with these materials. Phenolic resins may be attacked by the joint action of moisture, and alkali leached out from lime–soda glass, the bonding thus becoming weakened. With reinforced glass fibre products max. adhesion and the absence of voids are imperative. Epoxy resins adhere well to glass, but polyester resins do not; bonding may be effected by means of an "intermediary resin" such as Volan (methacrylatochromic chloride), which forms a bond with both the glass surface and the polyester resin, and this is applied from aq. soln. after the glass fibre has been cleansed by heat-treatment. Another, similar process is based upon vinyltrichlorosilane; a surface layer of a silicone complex is formed, having unsaturated vinyl groups which are free to combine with the resin.

Glass fibres based on borosilicate compositions are termed "E glass" in view of their suitability for electrical insulation. Lime–soda glass is useless for electrical applications, and it is seriously weakened by weathering, due to the leaching out of Na_2CO_3. Insulation products which may be used at very low temp. are produced by the accelerated weathering of soda–glass fibres, followed by the consolidation of the remaining, almost pure silica skeleton by suitable heat-treatment. The use of soda–lime glass in reinforced materials is limited by the fact that the glass does not withstand the heat cleaning treatment very well. The heat-treatment of woven glass fabrics releases the strains induced by the weave itself, and gives good draping quality, and softness of handle. The coloration of glass fibres is difficult, and the most satisfactory results have so far been obtained by using pigment resins to coat the fibres; if the coating is too heavy, the fibre is stiffened excessively. A limited range of fade-proof colours may be produced by the addition of metal oxides to the melt, during manufacture. Several specialised applications of glass fibres are briefly discussed; these include— air filtration, liquid filtration, and column packings for distillation and gas absorption work. J. W. D.

PATENTS

Acrylonitrile Fibres having Good Affinity for Acid and Basic Dyes. BrC. *BP* 730,032

Copolymers of acrylonitrile and derivatives of an $\alpha\beta$ mono-olefinic carboxylic acid containing the $>N{\cdot}N<$ group, e.g. maleic monohydrazide, are fibre-forming and have good affinity for acid and basic dyes. C. O. C.

Acrylonitrile Copolymers of Improved Affinity for Dyes. Chemstrand Corpn. *USP* 2,676,952

Copolymers of 85–99% of acrylonitrile and 15–1% of a monomer such as vinyl pyridine have their affinity for dyes increased by treating them with an alkyl halide so that the tertiary amino group is converted to a quaternary ammonium salt. C. O. C.

Solutions of Acrylonitrile Polymers. Dow Chemical Co. *BP* 729,382

Polymers containing $\leqslant$ 85% of acrylonitrile may be dissolved at moderate temperatures in an aqueous mixed brine containing $\geqslant$ 30% of a solvent salt but insufficient to dissolve the polymer and 5–25% of a non-solvent salt, the total of both salts being $\leqslant$ 55%. The solvent salts which may be used are $SnCl_4$ and the chlorides and nitrates of Zn, Ni, Co and Mn, the non-solvent salts being the chlorides and nitrates of Mg, Ca, Al and Na. Such solutions are useful for spinning tough fibres of the polymer. C. O. C.

Artificial Fibres from Polyacrylonitrile. Courtaulds. *BP* 729,472

Threads of polyacrylonitrile or a copolymer containing $\geqslant$ 20% of other polymerisable compounds, e.g. styrene, are prepared by dissolving the polymer in a mixture consisting essentially of water, a water-miscible diluent, e.g. methyl alcohol, and one or more highly water-soluble alkali metal or alkaline earth metal thiocyanates, guanidine thiocyanate, ammonium thiocyanate, LiBr, LiI, NaI, and $ZnCl_2$, which salt should constitute at least 35% of the solvent mixture. The solution is extruded into a coagulant bath consisting of an aqueous solution containing

the same salt and diluent mixture in roughly the same relative proportions as in the polymer solution and present in at least 10% volume concentration; the concentrations of salt and diluent in the bath are maintained below that at which spinning breaks down, which is about 50 solvent to 50 water. J. W. B.

Making and Colouring Felt (VIII p. 425).
Pigmenting Synthetic Linear Polymers before Melt Spinning or Extrusion (VIII p. 425).
Antistatic Action and Molecular Structure (X p. 427).
Heats of Wetting of Collagen, Leather, and other Organic and Fibrous Materials (XII p. 430).

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Cotton Quality Study. III— Resistance to Soiling. N. F. Getchell. *Text. Research J.*, **25**, 150–194 (Feb. 1955).

A comprehensive review of soiling with particular reference to cotton is presented under the headings— nature of soil, mechanisms of soiling, methods of measurement, soil-resistant treatments, problems for further research. There are 131 references. W. K. R.

Phase Changes in Soap–Oil Dispersions. B. W. Hotten and D. H. Birdsall. *Ind. Eng. Chem.*, **47**, 447–451 (March 1955).

The variation with temp. of the rate of oil filtration under vacuum has been found to be a sensitive indicator of phase change in greases. Typical soap–oil greases pass through three major structural types with rise in temp.— microcrystalline paste, gel, and solution. The nature of the soap cation has an important influence on the shape of the filtration–temp. curve; Ca and Al stearate dispersions gel and dissolve at lower temp. than Li and Na stearate dispersions. Unsaturation in the soap anion lowers gelation temp. The oil has little influence on the temp. of phase transition. W. K. R.

Detergency Studies with Sulphated Tallow Alcohols. L. Osipow, D. Marra, C. T. Snell, and F. D. Snell. *Ind. Eng. Chem.*, **47**, 492–496 (March 1955).

The effectiveness of detergents prepared by the sulphation of (*a*) saturated alcohols produced by the hydrogenation of tallow and (*b*) alcohols containing unsaturated groups produced by sodium reduction of tallow was evaluated under laboratory conditions. Both types are insufficiently soluble for use in liquid detergents. For washing both cotton and wool a 1 : 1 combination of the saturated and the unsaturated sulphates, in built formulations, compared favourably with commercial detergents. Without builders, the saturated sulphate was a better detergent for wool than the unsaturated sulphate. Data on the surface tension, interfacial tension, foaming power, and emulsifying ability of the two detergents, alone and in various built formulations, are tabulated. W. K. R.

Rôle of Carboxymethylcellulose in Detergency. H. Stüpel. *Melliand Textilber.*, **36**, 355 (April 1955).

Redeposition of soil is believed to be prevented by association of carboxymethylcellulose (C.M.C.) with dirt particles and with the fibre. It was found, however, that the normal red fluorescence of C.M.C. with Acridine Orange could not be observed on fibres washed in presence of the ether and the dye. S. R. C.

Optical Brightening Agents in the Bleaching of Wool. R. Keppler. *Textil-Praxis*, **10**, 284–286 (March 1955).

Optical brightening agents for wool are classified into three chemical groups— stilbene, benziminazole, and dibenziminazole derivatives. The affinity of optical brightening agents for wool, their fastness to light, washing, alkalis, and acids, and methods of detection and estimation on the fibre are described. B. K.

PATENT

Dry Cleaning of and Simultaneously Imparting Resistance to Gas Fading of Organic Derivatives of Cellulose. Celanese Corpn. of America. *BP* 729,170

Acetate rayon or the like is treated with a dry cleaning solvent containing 0·2–5·0% (in the solvent) of a gas-fading inhibitor. Excellent results are obtained when applying 2-amino-1-butanol by this means. C. O. C.

Adsorption of Available Chlorine and Quaternary Ammonium Compounds by Cotton and Wool Fabrics from Disinfecting Solutions (X p. 428).

VIII— DYEING

Variation of Micellar Size of Benzopurpurin 4B During the Dyeing of Cotton. S. R. Sivarajan, *J. Indian Inst. Science*, **A 36**, 282–292 (Oct. 1954).

The effect of salt and detergent on the micellar size of Benzopurpurin 4B during dyeing at 20° and 75°C. has been measured by means of light scattering experiments at 6500 A. (at this wavelength this dye does not absorb). It was found that in the presence of salt alone the micellar size remains unaltered during dyeing, the size being a function of salt concentration, whereas with detergent the micellar size alters considerably during dyeing. A change in absorption spectra in the presence of the detergent was also noted. W. J. M.

Felt Dyeing and Printing. Anon. *Dyer*, **113**, 505–507 (1 April 1955).

A practical review of methods employed in dyeing and printing felts. Acid and acid milling dyes are recommended for dyeing except where potting fastness of the dyed felt is required, and in this case chrome dyes are needed. Selected direct cotton dyes are suggested for printing felt except for blacks, where logwood is used, and very bright colours, where basic dyes are required. B. K.

Stability of Diazo Compounds. N. E. Fedorowa and E. I. Schelubjakowa. *Tekstil. prom.*, **14**, 36–39 (July 1954): *Textil-Praxis*, **10**, 281–284 (March 1955).

Investigations into the stability of diazo compounds have led to the following conclusions— (1) The diazo soln. of Fast Scarlet G base shows a high degree of stability in the presence of calcium chloride. (2) The least stability is shown with hydrochloric acid–diazo soln. with sodium acetate and sulphuric acid–diazo soln. with chalk, i.e. without stabilisers. (3) The maximum stability of diazo soln. derived from amines is achieved by the use of zinc oxide. (4) The properties of diazo soln. (printing pastes) which have been neutralised with chalk and zinc oxide are similar to those of dry more stable diazonium salts. B. K.

Metal-complex Dyes. G. Dierkes. *Rayonne*, **11**, 343–350 (March 1955).

A review of the chemistry and methods of application of premetallised dyes with special reference to the newer neutral-dyeing products. B. K.

Influence of High-temperature Dyeing on Tensile Strength of Fibres. F. Gasser and F. Weber. *Textil-Praxis*, **10**, 276–277 (March 1955).

The effects of high-temperature treatment of fibres under various conditions are detailed. With mixtures of natural and synthetic fibres it is preferable to dye the individual components separately if possible when using high-temperature techniques. B. K.

Ageing Damage of Viscose Rayon Staple Fibre dyed with Sulphur Black and subjected to Various Aftertreatments. G. Nitschke. *Melliand Textilber.*, **36**, 369–372 (April 1955).

The dyed goods should not be stored in warm moist conditions; rinsing should be thorough with the addition of a weak organic acid to the final water. If high drying temperatures or damp storage cannot be avoided, a weak alkali should be used in the final rinse. S. R. C.

Preliminary Study of the Dyeing of Cyanoethylated Cotton. F. Fordemwalt and R. E. Kourtz. *Text. Research J.*, **25**, 84–99 (Jan. 1955).

The dyeing of selected members of each class of dye on cyanoethylated cotton (approx. 3·5%N) has been examined. *Vat dyes* generally give heavier depths on cyanoethylated cotton than on untreated cotton, with similar light and wash fastness. Poor yarn and fabric penetration are improved by pigment padding or pigmentation followed by vatting. The yarn is unstable to caustic soda, which removes some cyanoethyl groups and hydrolyses some to carboxyethyl; nevertheless, by suitable control of dyeing, fibre damage can be kept within permissible limits. The fibre is stable under the conditions for applying other

classes of dyes. *Direct dyes*— By suitable selection a range of direct dyes with superior colour value on cyanoethylated cotton compared with cotton, equal wash fastness, and superior or equal light fastness is available. Aftertreatment with copper sulphate–hydroxylamine sulphate increases the light fastness of some dyes, making it higher than on cotton. *Diazotised and developed direct dyes*— The light fastness is equal or superior on cyanoethylated cotton to the same dyes on cotton. *Azoic dyes*— The colour value and the light fastness are superior on cyanoethylated cotton, while the wash fastness is equal to that on untreated cotton. *Sulphur dyes*— The colour value on cyanoethylated cotton varies from dye to dye, being in some cases weaker and in other cases higher than on cotton; wash fastness is equal and light fastness lower, but the latter is improved by aftertreatment with copper sulphate, potassium dichromate, and acetic acid. *Acid dyes*— The neutral-dyeing acid wool dyes have greater substantivities for cyanoethylated cotton than for cotton itself, but they are nevertheless poor. The cuprous ion technique gives higher colour values and light fastness. *Basic dyes*— On mordanted cyanoethylated cotton the colour value of basic dyes is much higher than on mordanted cotton, the fastness properties being similar. Unmordanted cyanoethylated cotton is generally dyed weaker than the mordanted material. *Disperse dyes*— Cyanoethylated cotton (3·5%N) is dyed weaker than acetate rayon, but the colour value increases as the N content increases. *Soluble vat dyes* yield pastel colours of good fastness. *Premetallised and chrome dyes* give much weaker dyeings (of good light and poor wash fastness) on cyanoethylated cotton than on wool.

R. F. A.

Dyeing Blends of Synthetic and Cellulosic Fibres. W. H. Hindle. *Amer. Dyestuff Rep.*, **44**, P191–P194 (14 March 1955).

Problems of dyeing blends of cellulosic and synthetic fibres are discussed and appropriate techniques described, with reference to blends containing not less than 25% of the new fibres. J. W. B.

Making and Colouring Felt. Anon. *Dyer*, **113**, 349–351 (4 March 1955).

A general review of felt manufacture including the types of raw materials used, mothproofing, dyeing, and finishing. B. K.

Dyeing Blends of Wool and Synthetic Fibres. H. Luttringhaus. *Amer. Dyestuff Rep.*, **44**, P194–P199 (14 March 1955).

Methods and recipes are given for dyeing blends of wool with silk, nylon, Vicara zein fibre, Dacron, Orlon, Acrilan, and dynel. Particular consideration is also given to dyeing solid colours on Vicara–nylon blends. Many of the colours are given Munsell notations for use with Munsell colour books. J. W. B.

Dyeing of Mixtures of Terylene Polyester Fibre and Wool. A. S. Fern and H. R. Hadfield. *J.S.D.C.*, **71**, 277–282 (June 1955).

This paper describes an investigation of the dyeing properties of Terylene–wool mixtures. Details are given of the dyeing variables which may affect the partition of dyes between Terylene and wool, and results are presented which enable the most suitable disperse dyes to be selected. A comparison has been made of the fastness properties of laboratory-dyed Terylene–wool mixture material with those of commercially piece-dyed wool. A suggested practical dyeing method for Terylene–wool mixtures is given. AUTHORS

Teddy Bear Pile Fabrics. Anon. *Dyer*, **112**, 1065–1067 (24 Dec. 1954); **113**, 35–37 (7 Jan.); and 115–117 (21 Jan. 1955).

Structure, wet processing, dyeing, and finishing of teddy bear fabrics consisting of mohair and various types of union materials, including the use of synthetic fibre piles such as nylon. B. K.

Colour Performance of Solution-dyed Acetate Rayon. A. F. Tesi. *Amer. Dyestuff Rep.*, **44**, P199–P201 (14 March 1955).

Data are presented showing that solution-dyed acetate rayon has excellent fastness to washing, rubbing, light, dry cleaning, perspiration, sea water, chlorinated water, and gas fumes. It also retains its tensile strength much better after prolonged exposure to sunlight than does uncoloured bright or dull acetate rayon. J. W. B.

Solvent Dyeing of Acetate Rayon. P. C. Duggan. *Amer. Dyestuff Rep.*, **44**, P202 (14 March 1955).

A brief survey is given of the relative advantages and disadvantages of solvent-dyeing acetate rayon either by using a 75% solution of methyl alcohol or by using a medium containing a mixture of organic acids with or without an addition of organic solvent and/or mineral acid. J. W. B.

Dye Binding to High Polymers. G. Oster. *J. Polymer Sci.*, **16**, 235–244 (April 1955).

Examples are presented showing that cationic dyes bind to polymeric acids by simple electrostatic forces. Competitive binding for the substrate is illustrated by the addition of hydrogen ions, calcium ions and streptomycin. Anionic binding to uncharged or negatively charged substrates is attributed to hydrogen bonding with carbonyl groups of the substrate. Van der Waals forces are also important in binding. Examples are given for the substrates native serum albumen, denatured ovalbumen, polyvinyl pyrrolidone and water soluble urea–formaldehyde resin. A new fluorimetric technique for following the binding of anions is described. Changes in spectral and photochemical properties of dyes following binding are summarised. W. R. M.

PATENTS

Pigmenting Synthetic Linear Polymers before Melt Spinning or Extrusion. FBy. *BP* 729,584

In the mass pigmenting of synthetic linear polymers agglomeration of the pigment can be avoided by producing a polymer containing a high proportion of pigment, which polymer has a much lower degree of polymerisation than the polymer to be melt spun. The two polymers need not be made from the same monomers. Adding a suitable amount of the pigmented lower polymer to that to be melt spun gives uniform distribution of the pigment without agglomeration. C. O. C.

Fast Dyeings or Prints on Cellulosic Fibres with Metallisable and/or Metalliferous Dyes. S. *BP* 728,071

The fibres are treated during or after dyeing or printing in an alkaline medium containing a low molecular complex compound of a polyvalent metal and ammonia or low molecular amine. The dyed or printed fibres are also treated with the water-soluble condensate obtained by heating a di- or tri-alkylene polyamine (alkylene of 2–3 C) with cyanamide or dicyandiamide. Suitable low molecular complex compounds of polyvalent metals comprise those with ammonia, low molecular amines or hydroxy carboxylic acids, e.g. glycollic or tartaric acid. C. O. C.

Continuous Fixation of Dyes on Cloth. Etablissements Schaeffer & Cie. *BP* 729,353

The cloth is impregnated with a single direct dye or with several leuco vat dyes and is then passed between a heated imperforate surface and a liquid- and gas-tight blanket firmly applied to the surface so as to allow no loss of heat or steam from the fabric and provoke fixation of the dye in the fibres. C. O. C.

Vat or Sulphur Dyeing in Molten Metal Machines. Standfast Dyers & Printers. *BP* 728,138

When using liquors containing hydrosulphite in a molten metal machine attack by the hydrosulphite decomposition products on the molten metal with consequent sludge formation is avoided by adding $NaNO_3$ to the liquor. C. O. C.

Dyeing Mixtures of Wool and Polyester or Organic Derivative of Cellulose Fibres. Celanese Corpn. of America. *BP* 728,271

The materials are dyed in a bath containing a wool dye, a disperse dye, an anionic dispersing agent, e.g. Tamol N, and a non-ionic dispersing agent, e.g. polyvinyl alcohol. In dye liquors containing these dispersing agents the disperse dye does not stain the wool to the same extent that it would in their absence. C. O. C.

Dyeing Plastic Sheets for Use in Glare Screens (I p. 413).
Diazoamino Compounds for Production of Azoic Dyeings and Prints by Neutral Steaming (IV p. 418).
Application and Limitations of Neutrogen Dyes (IX below).
Printing and Dyeing (IX below).
Printing or Padding Fibrous Sheet Material (IX below).
Dyed Aminised Cotton Fabrics of Low Combustibility (X p. 428).

IX—PRINTING

Prints and Dress [17–19th centuries]. C. Zuber. *Teintex*, **20**, 270–294 (April 1955).
An essay on the mechanics of printing related to the fashions of the period. 23 illustrations. S. R. C.

Application and Limitations of Neutrogen Dyes. M. Binder. *Teintex*, **20**, 255–270 (April 1955).
Although the pastes are somewhat unstable, e.g. to reduction, this disadvantage is claimed to be outweighed by the possibility of neutral steaming. Joint colouring with pigments and vat, direct, and chrome dyes is discussed. S. R. C.

PATENTS

Applying Dyes by a Sewing Machine. A. J. Hall. *USP* 2,675,755
The needle dips into dye liquor after passing through the material, the eye collects some of the liquor which is removed by the material when the needle returns through it. C. O. C.

Hardening the Dye-absorptive Hydrophilic Colloidal Layer of Black Cinematograph Films. Technicolor Motion Picture Corpn. *USP* 2,676,116
When the gelatin layer is heated with an aqueous solution of a chromic salt, e.g. chromic sulphate or chrome alum, containing both the purple and green forms of chromium, the purple form hardens the gelatin whereas the green form acts as a mordant for the dye in subsequent imbibition transfers without hardening the gelatin. This may be made of practical use by using a pH 3–4 chromic salt containing 25–70% of the green form of chromium and 75–30% of the purple form. After thorough rinsing the hardened film is ready for imbibition printing. C. O. C.

Subtractive Colour Photography. Kodak. *BP* 728,769
A method of subtractive colour photography using coloured colour couplers. C. O. C.

Colour Photography involving No Colour Distortion. E. Gretener. *BP* 730,050

Subtractive Three-colour Method of Colour Photography. S. Papadakis. *BP* 730,122

Colour Filter and Apparatus for Determination of Printing Filters in Colour Photography. AGFA A-G für Photofabrikation. *BP* 729,004

Decorating Ceramic Bodies. Ceratile Corpn. *BP* 729,135
A method of applying a glazed design followed by a second glaze which glazes after glost firing but will not fuse or run into the design glaze but tend to separate so as to produce unusual effects, although predetermined, in the glaze decoration. C. O. C.

Printing and Dyeing. FBy. *BP* 728,655
Prints and dyeings of equal fastness to those obtained with pigments and of improved fastness to rubbing and evenness are obtained by using aqueous pastes containing dyes which are insoluble in water but soluble in the binder used. The binder is preferably a water-soluble polymer which is insolubilised in the fibre by treating it with a cross-linking agent, e.g. a polymeric polyamine treated with triacrylformal. C. O. C.

Printing or Padding Fibrous Sheet Material. FBy. *BP* 729,119
A printing paste is used containing a vinyl polymer which contains no reactive H atoms and no reactive quantitatively exchangeable halogen atoms and is treated on the fibre with a cross-linking agent. Suitable polymers are those of methyl methacrylate, butadiene, *N*-vinyl-2-pyrrolidone, etc. and suitable cross-linking agents are hexahydro-1:3:5-triacrylyl-*s*-triazine, an acrylic acid amide of a polyamine, an acrylate of a polyalcohol or a polyester from a polyfunctional unsaturated acid.

BP 729,122
Prints of extremely good fastness to rubbing and washing are obtained by use of an emulsion of a vinyl or divinyl polymer or copolymer and a high molecular basic compound and, if the basic compound is not a cross-linking agent, treating the prints with a compound containing ≥ 2 groups capable of reacting with the high molecular compound. Suitable polymers are those mentioned above and suitable basic compounds comprise basic polyureas, basic polyamides especially polyoxamides, basic polyurethanes, etc. C. O. C.

Mordants for fixing Dyes in Photographic Layers (III p. 414).
Diazoamino Compounds for Production of Azoic Dyeings and Prints by Neutral Steaming (IV p. 418).
Felt Dyeing and Printing (VIII p. 424).
Stability of Diazo Compounds (VIII p. 424).
Fast Dyeings or Prints on Cellulosic Fibres with Metallisable and/or Metalliferous Dyes (VIII p. 425).

X—SIZING AND FINISHING

Use of Dieldrin to protect Wool against Insect Attack. M. Lipson and R. J. Hope. *Nature*, **175**, 559–560 (2 April 1955).
Dieldrin, applied from petroleum-ether soln. or aqueous suspension, gives a satisfactory mothproofing effect on wool which withstands repeated washing and dry-cleaning treatments. A. J.

Improvement of Lustre of Cotton. X—Special Swelling Treatments and Non-thermosetting Additives. L. Fourt and H. J. Elliott. *Text. Research J.*, **25**, 11–19 (Jan. 1955).
Excessive swelling produced by strong sulphuric acid, zinc chloride, and probably caustic soda below 0°C. decreases lustre because the fibre surface is roughened. Drastic shrinkage of yarn volume, combined with film formation and disappearance of fuzz, can leave the fabric very sheer and transparent, attributes often correlated with lustre. Mechanical action on the plastic cellulose, or on films of cellulose or hydroxyethylcellulose deposited from solution, can give increased lustre through flattening and mechanical stabilisation in the flattened form. These cellulose-affecting treatments tend to leave the fabric stiff, if they go beyond normal mercerising. Other additive finishes act on lustre either through film formation or by stabilising mechanical effects and controlling fabric structure. The effect of the more thermoplastic resins together with mechanical smoothing is less than that of mechanically smoothing the untreated fabric, so the chief advantage of the resin finish lies in effects such as possible greater stability of a mechanical finish with the resin present. In the higher ranges of lustre the eye can distinguish between that produced by film formation or flattening, and lustre arising from yarn and fabric structure and from mercerising. R. F. A.

New Cotton Properties through Chemical Research. C. H. Fisher. *Text. Research J.*, **25**, 1–11 (Jan. 1955).
A review of chemical methods of imparting durable effects to cotton is made and some possible developments are discussed. A list of some chemical modifications of cotton studied by the Southern Regional Research Laboratory is given. The following topics are discussed—(*a*) the effect of changes in polymer structure (i.e. crystallinity, association of polymer chains, cross-linking, and side-chains) upon the physical properties of cotton textile materials; (*b*) the effects of changes in fibre dimensions, produced, e.g. by mercerisation, on lustre, slipperiness, and resistance to clinging and to soiling; (*c*) the effect of alterations in the hydrophobic properties of cotton, produced by reaction with, e.g. aralkyl chlorides, on electrical properties and behaviour towards moisture. Similarly, the modified resistance of cotton to deterioration by light, oxidation, mildew, and heat achieved by the attachment of chemical groups either to the cellulose chain or to adjacent material is discussed. There are 32 references. R. F. A.

Special Elastic Properties of Cotton Yarn and Cloth mercerised without Tension. C. F. Goldthwait and A. L. Murphy. *Text. Research J.*, **25**, 47–57 (Jan. 1955).
Suitable cotton yarn or cloth mercerised without tension shrinks and its elastic properties are changed. This fact

is used in the successful commercial production of a semi-elastic and conforming cotton gauze bandage. R. F. A.

Pilot-plant Production and Properties of Cyanoethylated Cotton. J. Compton, W. H. Martin, B. H. Word, and R. P. Barber. *Text. Research J.*, **25**, 58–75 (Jan. 1955).

The morphological and molecular structure of cotton is discussed in some detail. A procedure for the cyanoethylation of a cotton package by pretreatment with 1·5% NaOH soln. for 15 min. followed by treatment for 1 hr. at 135°F. with acrylonitrile is described. The treatment of loose fibre and cotton cloth is also described; a continuous process for cotton fabric has been operated. The half-time of reaction of pretreated cotton with acrylonitrile for 3–4% N content is 10–20 min. Cyanoethylation increases the fibre diameter in proportion to the N uptake; it reduces the moisture regain to 5% at 3–4% N content. Breaking strength and elongation of cotton yarn and breaking strength of fabric are increased, but the increase is not related to the N content. The resistance to microbiological attack of cyanoethylated cotton is complete if the N content is $> 3{\cdot}5\%$, and is very much higher than that of cotton at lower N values. Resistance to heat and acids is also increased by cyanoethylation. The flat abrasion resistance increases with increasing N content; flex abrasion resistance increases up to 2·5% N, thereafter decreasing. Provided that chlorine is absent, there is no loss in strength on washing; in the presence of chlorine cyanoethylated cotton loses strength during washing at the same rate as cotton itself. Cyanoethylated cotton is generally more receptive to dyes of all classes, and the dyed fibre has fastness properties equal to those of untreated cotton. 44 references. R. F. A.

Progress Report on Cyanoethylated Cotton. J. N. Grant, L. H. Greathouse, J. D. Reid, and J. W. Weaver. *Text. Research J.*, **25**, 76–83 (Jan. 1955).

The mechanism of the cyanoethylation of cotton is discussed, and it is shown that for best efficiency the pick-up of sodium hydroxide must not exceed 15% of the weight of the cotton. Measurements of physical properties show that any increases in the breaking strength or elongation of yarns are results of changes in frictional forces. The physical properties of cyanoethylated fabrics reflect the properties of the yarn and also depend on fabric construction. Extremely high resistance to flat abrasion is obtained with high cyanoethyl substitution. The density of cyanoethylated cotton is shown to be linearly related to the N content, and the spread of values obtained has been found to give an estimate of the uniformity of treatment. Cyanoethylated cotton can be dyed by methods developed for polyacrylonitrile fibres, and the intensity of colour can be taken as a measure of the amount of cyanoethylation. R. F. A.

Insolubility in Cuprammonium Hydroxide as a Means of detecting Cross-linking in chemically Modified Cotton. W. A. Reeves, G. L. Drake, O. J. McMillan, and J. D. Guthrie. *Text. Research J.*, **25**, 41–46 (Jan. 1955).

Native cellulose dissolves in cuprammonium hydroxide solution, but when cross-linked becomes a space polymer and is insoluble in this reagent. In all cases the cellulosic derivatives made from cellulose and compounds that could cross-link it by virtue of two reactive terminal groups, were partly soluble in cuprammonium hydroxide. Compounds incapable of cross-linking cellulose produced derivatives soluble in cuprammonium hydroxide. Cross-linking compounds were formaldehyde, glyoxal, chloroethylsulphuric acid, $\alpha\gamma$-glycerol dichlorohydrin, and a mixture of $\alpha\beta$- and $\alpha\gamma$-glycerol dichlorohydrin, tetrakishydroxymethylphosphonium chloride, compounds containing two or more *N*-hydroxymethyl groups, hexamethylene di-*iso*cyanate, vinyl sulphone, 4-disulphato-2-butyne, and potassium (disulphatoethyl) amine. Aminised cotton (2-aminoethylcellulose), which is soluble in cuprammonium hydroxide, was made insoluble by cross-linking it with either formaldehyde or tetrakishydroxymethylphosphonium chloride. R. F. A.

Crease-resistant Finishes on Cotton. H. Y. Jennings. *Text. Research J.*, **25**, 20–24 (Jan. 1955).

The importance of residual alkali on the acid polymerisation catalyst and of mixed polymers which have better properties than either of their constituents is discussed. Increasing crease-resistance of cotton, while conferring thermoplastic properties on the fabric, renders making-up difficult and hence is no alternative to thermoplastic synthetic fibres. The Monsanto and T.B.L. systems of crease-resistance measurement are discussed— the latter is claimed to correlate better with field trials. R. F. A.

Controversial Aspects of Crease Resistance of Cellulose Fabrics. A. C. Nuessle, M. N. Fineman, and E. O. J. Heiges. *Text. Research J.*, **25**, 24–40 (Jan. 1955).

Some controversial questions are discussed— (*a*) differences between cotton and rayon and the reasons therefor; factors considered include tensile strength, tear strength, abrasion resistance, permeability of the fibres, and acid hydrolysis; (*b*) a brief commentary on attempts to make cotton more like rayon; (*c*) mechanism of crease recovery, with emphasis on the question of covalent cross-linking *versus* resin formation; (*d*) other aspects of crease resistance emphasising subjective characteristics such as fabric handle. R. F. A.

Application of Silicone Water-repellents to Cotton. A. A. Cook and N. C. Shane. *Text. Research J.*, **25**, 105–110 (Jan. 1955).

The application of a silicone water-repellent, Hydro-Pruf (Arkansas Co.), together with a curing catalyst (Cobelfix) and the curing at 320°F. for 5–8 min. are described. The durability of the finish to dry cleaning and washing and the variation in fastness with increasing silicone content are described. Thermosetting resins (methylated melamine–formaldehyde) increase the durability of the silicone finish. The treatment of cotton with Hydro-Pruf reduced the tensile strength, but the reduction is not regarded as excessive. The flex abrasion resistance is greatly increased by the silicone treatment; when resins are used together with the silicone finish the abrasion resistance is higher than with resin alone. R. F. A.

Flame-resistant Cotton Fabrics—Emulsion Treatment using an Organic Phosphorus-Bromine Polymer. J. G. Frick, J. W. Weaver, and J. D. Reid. *Text. Research J.*, **25**, 100–105 (Jan. 1955).

Flame-resistant cotton fabrics have been prepared by treatment of the fabric with an aqueous emulsion of an organic polymer, prepared from bromoform and triallyl phosphate. Recipes for the laboratory preparation of 33% and 40% emulsions are given. The emulsion is padded in conventional equipment on to the fabric, which is dried (at 80–110°C.) and cured (4–6 min. at 140°C.) in a ventilated oven. The finish is durable to repeated laundering, and when applied with proper plasticisation to fabrics of 8–9 oz. weight does not materially change other textile properties. R. F. A.

Washable Rayons. I. H. Welch. *Amer. Dyestuff Rep.*, **44**, P216–P219 (28 March 1955).

A detailed description is given of the Avcoset rayon stabilisation process, which, it is claimed, gives durable shrinkage control, good handle and colour retention, freedom from seam-fraying, and greatly reduced fibrillation. J. W. B.

Antistatic Action and Molecular Structure. G. R. Ward. *Amer. Dyestuff Rep.*, **44**, P220–P226 (28 March 1955).

Mechanisms of charge generation and dissipation are discussed, and a method of calculating the conductivity needed to counteract electrostatic effects on yarns and fabrics is proposed. Bulk resistivities are calculated for experimental oxyethylated nonylphenols, and the influence of hygroscopicity and of oxyethylation molar ratio is studied. Similar measurements made on acetate rayon and nylon fabrics treated with an oxyethylated alkylphenol indicate that several factors, such as viscosity, dielectric constant, and ionic solvation, are involved as well as moisture content. An increase from 35% to 70% R.H. leads to a tenfold increase in conductivity. Evaluation of static charge is carried out by squeezing samples in a Neoprene glove, dropping them into a metal beaker, and measuring the charge by means of an electrometer and an oscilloscope. Experiments include a study of the effect of storage, and are made on acetate rayon, nylon, X-51, Acrilan, Orlon, dynel, and Dacron. Loss of efficiency due to storage is considered to be caused by diffusion of the

antistatic into the fibre, and increasing size and complexity in the hydrocarbon portion is found to give better properties in this respect; oxyethylation appears to be helpful apart from the molecular size factor. J. W. B.

Adsorption of Available Chlorine and Quaternary Ammonium Compounds by Cotton and Wool Fabrics from Disinfecting Solutions. M. T. Goldsmith, M. A. Latief, J. L. Friedl, and L. S. Stuart. *Appl. Microbiol.*, **2**, 360–364 (1954): *Chem. Abs.*, **49**, 5849 (28 April 1955).

Cotton needed 8–20 p.p.m. and wool 41–800 p.p.m. of available Cl of aq. NaOCl to disinfect 3 g. of fabric inoculated with *Escherichia coli* and *Micrococcus pyogenes var. aureus* 209P using 100 ml. of the germicide. The wool was damaged by the Cl. Cotton was disinfected with 200–2000 p.p.m. of alkyldimethylbenzylammonium chloride (Alk of 8–18 C) (I); wool needed 100–666 p.p.m. Cotton adsorbed 70–90% of the available Cl when 10–30 p.p.m. soln. were used while wool adsorbed practically all the Cl from soln. containing 800 p.p.m. In low concentrations of I cotton adsorbed up to 75%. Wool removed 25–75% of I from soln. of 10–100 mg./100 ml. Stronger soln. are required to disinfect fabrics contaminated with *M. pyogenes* than to disinfect those contaminated with *E. coli*. C. O. C.

Preparation of Microbiologically Resistant Wool by Chemical Modification.

III—Reaction with Monofunctional Compounds. H. Zahn and A. Würz. *Text. Research J.*, **25**, 111–114 (Feb. 1955): *Melliand Textilber.*, **36**, 121–123 (Feb. 1955).

A soil burial test was used to evaluate the resistance of wool yarn to microbiological attack, and gave reproducible results. Chemical modification of the wool by refluxing with acetic anhydride–acetic acid or with *p*-toluenesulphonyl chloride in acetone–water gave good microbiological resistance, but treatment with other monofunctional acylating agents was ineffective. Alkylation of wool with epichlorohydrin or benzyl chloride caused only slight improvement in resistance.

IV—Reaction with 1-Fluoro-2:4-dinitrobenzene. H. Zahn and A. Würz. *Text. Research J.*, **25**, 115–118 (Feb. 1955): *Melliand Textilber.*, **36**, 123–125 (Feb. 1955).

Microbiologically resistant wool is obtained by treatment with 1-fluoro-2:4-dinitrobenzene from either alcohol or water at 40°C., provided sufficient is used. Thus with ca. 50% (on wt.) the treated wool withstands $>$ 100 days soil burial, but smaller quantities are less effective. At 60–100°C. the time of treatment can be reduced appreciably. No improvement in resistance is obtained by similar treatment with 1-chloro-2:4-dinitrobenzene at 40°C., but application of ca. 50% of this compound at 100°C. gives good resistance.

V—Reaction with Bifunctional Compounds. H. Zahn, A. Würz, and A. Räuchle. *Text. Research J.*, **25**, 118–120 (Feb. 1955): *Melliand Textilber.*, **36**, 125–126 (Feb. 1955).

Wool is made microbiologically resistant by treatment with 1-fluoro-5-chloro-2:4-dinitrobenzene, 1:5-difluoro-2:4-dinitrobenzene, 1:3-bischloromethyl-4:6-dimethylbenzene, and *p*-bromo-ω-bromoacetophenone, provided that sufficient of each reagent (ca. 50% on wt. of wool) is used.

VI—Quinone, Ninhydrin, and Heavy-metal Salts. H. Zahn, A. Würz, and A. Räuchle. *Text. Research J.*, **25**, 120–124 (Feb. 1955): *Melliand Textilber.*, **36**, 126–128 (Feb. 1955).

Treatment with benzoquinone in concn. $>$ 0·5% soln. at 100°C. and with ninhydrin makes wool highly resistant to microbiological attack. Moderate resistance is provided by impregnation with 2:4-dinitrophenol, treatment with mercuric acetate, chroming, and chrome dyeing. W. K. R.

Deposition of Polyacrylonitrile in Wool. I—Reaction Mechanism. L. Valentine. *J. Textile Inst.*, **46**, T270–T283 (April 1955).

Acrylonitrile is polymerised inside wool fibres using the Fe^{++}–H_2O_2 technique. It is shown that both the rate of deposition and the saturation weight increases are increased by raising temperature, or concentration of acrylonitrile, but both are independent of H_2O_2 concentration over the range 0·001–0·01%. Up to a weight increase of 40% the weight of polymer formed internally is directly proportional to the square root of the time of treatment; above this and up to a saturation value the rate decreases. Results are very fully discussed; the behaviour is characteristic of a diffusion-controlled system, and data are analysed in terms of an apparent diffusion coefficient which may correspond to the diffusion of acrylonitrile into wool. J. W. B.

Heat-setting of Synthetic Fibres—IV. F. Fourné. *Textil-Praxis*, **10**, 270–275 (March 1955).

Methods of heat-setting synthetic fibres in various forms are described in detail. Despite various difficulties in application, setting with saturated steam gives more reliable results than hot air fixation and is therefore preferred by many processors of synthetic fibres. B. K.

PATENTS

Dyed Aminised Cotton Fabrics of Low Combustibility. U.S. Secretary of Agriculture. *USP* 2,695,833

The fabric is aminised to an amine N content of $<$ 0·6% and then dyed with an acid-reacting azo dye containing at least one phosphato and sulpho radical so that $<$ 7% of the weight of the dyed fabric is dye. The dyed fabric has low combustibility. If the dyed fabric is impregnated with a solution of poly(triallyl phosphate) until $<$ 10% of the polymer is taken up by the fabric, which is then dried and cured, a flame-resistant finish is obtained which is fast to washing and which satisfies the A.A.T.C.C. Standard Test Method 3437 Vertical Flame Test. C. O. C.

Coating Fibres with the help of Gas Agitation. F. D. Farnam Co. *USP* 2,676,099

The fibres are formed into a dilute aqueous pulp which is placed in a cylindrical vessel whose side walls curve into its bottom. Here it is treated with air jets so as to agitate the whole mass and prevent agglomeration of the fibres. During agitation the coagulant is added and when this has been completely adsorbed by the fibres an emulsion of the coating agent is added so that it immediately disperses into the agitated slurry. Finally a second addition of coagulant is made to clear the liquor from which the fibres are separated in any desired manner. C. O. C.

Artificial Leather. H. Eberle and O. Saladin. *BP* 730,221

A fibrous support is covered with a dispersion of an olefinic polymer containing 5–30% of water. It is dried at $>$ 120°C. so that a network of pores is produced in the coating. If desired the resinous coating may be gelatinised and grained. C. O. C.

Non-woven Fabric. DuP. *USP* 2,676,128

A non-woven fabric is composed of non-fusible fibres forming the fabric structure and bonded together by fusible, elastic polyester fibres obtained by copolymerising compounds of formula $HOOC{\cdot}CH_2XCH_2{\cdot}COOH$ (X = linear chain of 4–9 atoms of which $<$ 4 are O atoms the remainder being hydrocarbon C atoms any two O atoms being separated by $>$ 1 C atom, there being $\geq$ 3 hydrocarbon C atoms as side chain substituents) with terephthalic and/or bibenzoic and/or ethylene bis *p*-oxybenzoic and/or tetramethylene bis-*p*-oxybenzoic and/or 2:6-naphthalic acid and with a compound of formula $HO(CH_2)_nOH$ (n = 2–6) e.g. fibres of the polyester containing 40% ethylene glycol sebacate and 60% ethylene glycol terephthalate. C. O. C.

Antistatic Treatment of Non-cellulosic Hydrophobic Fibres. DuP. *USP* 2,676,122

Fibres such as nylon, Terylene, polyacrylonitrile fibres, etc. are given an excellent antistatic finish by treating them with mixtures of compounds of formula—

$$(Alk\ O)_x{\cdot}PO{\cdot}[O(NX)H]_y$$

(x = 1 or 2; $y = 3 - x$; Alk = *n*-Alk of 8–16 C; NX = one of the three ethanolamines or the three ethanolmethylamines, or morpholine), e.g. an equimolecular mixture of the bis-diethanolamine salt of monolauryl phosphate and the diethanolamine salt of dilauryl phosphate. C. O. C.

Applying Antistatic Agents to Dielectric Materials. Chicopee Manufacturing Corpn. *USP* 2,676,115

Scraping dielectric materials, e.g. Saran, immediately before and particularly during the application of the antistatic agent gives greatly improved antistatic property with startling reduction in the amount of the agent needed. C. O. C.

Sizing Polyethylene. DuP. *USP* 2,676,120

An aqueous composition containing dispersed polyvinyl acetate, an alkali metal alkyl sulphate (Alk of > 7 C), $ZnCl_2$ and, if desired, NaCl is an excellent antistatic size for polyethylene.

USP 2,676,121

An aqueous composition containing dispersed polyvinyl acetate, LiCl and an alkali metal or ammonium salt of wood rosin or of hydrogenated or polymerised wood rosin, is used. C. O. C.

Imparting to Glass Fibres Better Adhesion to Synthetic Resins. Owens-Corning Fiberglas Corpn. *BP* 729,452

Treatment with a water-soluble salt of an unsaturated polysiloxanol of formula—

$$\begin{matrix} Z & Z \\ | & | \\ R\cdot Si\cdot(O\cdot Si)_n\cdot OM \\ | & | \\ Z & R \end{matrix}$$

(M = Na, K, Li or NH_4; R = same or different, but one, preferably all, must be an unsatd. organic radical of < 6 C in a straight chain; Z = OM or R) imparts to glass fibres improved adhesive properties for synthetic resins. C. O. C.

Unsaturated Polyester Resin–Glass Fibre Laminates. Union Carbide & Carbon Corpn. *BP* 728,264

Heat cured unsaturated polyester–glass fibre laminates of improved wet strength are obtained by sizing the glass fibres, before applying the resin, with 0·05–2·0% (on wt. of the glass) of a vinylpolysiloxane resin. C. O. C.

Treating Textiles with a Colloidal Solution of Silica. Monsanto. *BP* 728,237

If a textile of any type is treated with an aqueous colloidal solution of silica in presence of a water-soluble aluminium, ferric, chromic or ceric salt then the silica exhausts on to the fibres. C. O. C.

Crease-resisting Finish for Regenerated Cellulose Fibres. Phrix-Werke. *BP* 727,919

Modification of *BP* 710,441 (J.S.D.C., **70**, 376 (1954)). An excellent crease-resisting finish of very good fastness to washing is obtained if boric acid or a borate is added to the impregnating solution which should have pH 3·0–4·2. C. O. C.

Rendering Regenerated Cellulose Fabrics Resistant to Wet Creasing. Tootal, Broadhurst Lee Co. *BP* 727,881

Fabric made wholly or partly of regenerated cellulose is rendered resistant to wet creasing by treating it with steam at > 10 lb./sq. in. above atmospheric pressure. The treated fabrics have less need for ironing or pressing after washing.

BP 727,882

Fabric made wholly or partly of regenerated cellulose is treated with a swelling agent and given a crease-resisting finish. This yields a fabric which—(*a*) on wetting loses any dry crease marks it may have acquired, and (*b*) is resistant to wet creasing so that it does not need ironing or pressing after washing.

BP 727,888

Fabric made wholly or partly of regenerated cellulose and free from crease marks is treated in open width with a swelling agent so that the regenerated cellulose swells without substantial damage. The fabric is kept in contact with the swelling agent while in open width and free as possible from tension, until it has been rendered crease-shedding and wet crease-resisting. Finally a setting effect is imparted by condensing within and not between the fibres just enough of a thermosetting synthetic resin or by treating with an aldehyde in presence of acid to impart to the fabric a shrinkage of 0% in weft and warp when given the test described in *The Fibro Manual*, p. 239 (London, 1949).

BP 727,889

Fabric made wholly or partly of regenerated cellulose can be made crease-shedding and wet crease-resisting by treating it with a swelling agent for regenerated cellulose in absence of crease marks and tension.

BP 727,890

Fabric made wholly or partly of native cellulose can be treated with a swelling agent so that the treated fabric will when wet, lose its crease marks and so need not be ironed or pressed after washing. C. O. C.

Treating Fabrics with Resinous Condensates to Impart Resistance to Abrasion. Tootal Broadhurst Lee Co. *BP* 728,440

The resistance to abrasion of a fabric composed wholly or partly of regenerated cellulose and containing a thermosetting amino-aldehyde resin in the fibres can be improved by treating it in superheated steam in presence of acid after baking to form the resin. C. O. C.

Laminated Fabrics. Trubenised (Gt. Britain). *BP* 727,933

When making laminated fabric with the aid of a thermoplastic material contraction of the threads of such material is avoided by using for the thermoplastic thread a mixed yarn spun from fibres of low m.p. material which contracts on heating and fibres of a second, non-thermoplastic material which does not contract on heating. The non-thermoplastic fibres interlock with one another to form a yarn independently of the thermoplastic fibres which must be so much shorter than the non-thermoplastic fibres that their contraction on heating is not imparted to the non-thermoplastic fibres. C. O. C.

Multiply Fabric. Bradford Dyers' Assocn. *BP* 727,591

Bonding together of the plies is at least initiated and the assembly compressively shrunk in a single operation by the combined application of heat and pressure, e.g. by clamping the fabric firmly to a surface which is caused to contract so that the fabric simultaneously contracts and never recovers its original length. C. O. C.

Shrinking of Collars and other Fabric Articles. Bradford Dyers' Assocn. *BP* 727,569

When collars or other stitched articles having parts differing in thickness from one another are shrunk on a machine of the type described in *BP* 402,087 (J.S.D.C., **50**, 114 (1934)), creasing or puckering can be avoided by appropriate selection of the rubber or like material of the belt and by appropriate transmission of the drive to the moving parts. The material of the belt must be such that the pressures applied by it to the parts of different thickness are proportional to those thicknesses. C. O. C.

Fabrics composed of Adhesively-bonded Fibres. Carl Freudenberg. *BP* 728,865

A porous, crease-resisting, shrink-proof fibrous sheet material is produced by placing several carded laps each 0·1–1·2 mm. thick and weighing 15–35 g./sq.m. in layers and then uniting them by pressure and heat with slight stretching to form a loose fleece weighing 40–400 g./sq.m. At least one surface of the fleece is then treated with a liquid to produce slight surface cohesion, e.g. by applying an adhesive in solution and then drying so as to deposit 3–10 parts by weight of adhesive per 100 parts of fibres. An aqueous binder is then pressed as a stable flowable foam into the fleece, so as to produce complete and uniform penetration with simultaneous destruction of part of the foam cells. This is done so as to incorporate 20–140 parts (solid wt.) of binding per 100 parts of fibres. The impregnated sheet is then dried in absence of pressure. C. O. C.

Non-woven Floor Coverings. Bigelow-Sanford Carpet Co. *BP* 728,455

A continuous length of soft surfacing material is advanced a predetermined distance longitudinally of and spaced from an adhesive-coated backing sheet to form a fold in the material extending away from the sheet. A bight in the material is formed longitudinally of the sheet and this bight is pressed against the adhesive and is caused to adhere to it by local setting of the adhesive. The backing sheet is then advanced with the adhering soft surfacing material. C. O. C.

Photodegradation of Cellulose, particularly in the Presence of Titanium Dioxide (VI p. 422).
Dry Cleaning of and Simultaneously Imparting Resistance to Gas Fading of Organic Derivatives of Cellulose (VII p. 424).
Making and Colouring Felt (VIII p. 425).
Printing or Padding Fibrous Sheet Material (IX p. 426).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Magnetic Resonance Absorption of Protons in Water adsorbed on Carbon and Cellulose. K. Tanaka and K. Yamagata. *Bull. Chem. Soc. Japan*, **28**, 90–92 (Jan. 1955).

The line shapes of proton magnetic resonance absorption in water adsorbed on carbon and on cellulose indicate that on carbon water is firmly adsorbed as a monolayer above which protons are mobile, whereas with cellulose it is likely that water is adsorbed in multilayers. A. J.

Acetylation of Cellulose in presence of Phosphoric Acid. Z. A. Rogovin, N. Chernÿsheva, and A. A. Konkin. *Khim. prom.*, 232–233 (1954): *Chem. Abs.*, **49**, 1321 (25 Jan. 1955).

A study is made of factors affecting the course of the acetylation of cellulose, and of the reverse, saponification reaction, in homogeneous and heterogeneous conditions in presence of phosphoric acid. A. E. S.

ω-*p*-Aminoacetophenone Ether of Cellulose. R. R. McLaughlin and D. B. Mutton. *Canadian J. Chem.*, **33**, 646–656 (April 1955).

Seven forms of cellulose or cellulose derivatives have been treated with ten different reagents under varying conditions with the aim of preparing an ether containing diazotisable amino groups. Reasons are given why only one reagent, under a given set of conditions, gave a product with a substantial degree of substitution and satisfactory physical properties. This product, the ω-*p*-aminoacetophenone ether of cellulose, was prepared by reaction between ω-chloro-*p*-aminoacetophenone and ethanol-washed soda-cellulose. It can be diazotised and coupled with the usual reagents to give cellulose-azo colouring matters, xanthated and spun as rayon filament, dyed directly with acid dyes, rendered organo-soluble by nitration, and carboxymethylated after coupling to give coloured water-soluble derivatives. It is insol. in the usual cellulose solvents, and as stable as cellulose to acid and alkaline hydrolysis. W. R. M.

PATENT

Cellulosic Plastic Compositions capable of being dyed with Acid or Metalliferous Dyes. Société des Usines Chimiques Rhône-Poulenc. *BP* 729,183

Dispersing a cellulose ether or ester and a polymeric aminoethylvinyl ether in a liquid so as to form a true solution or fine suspension and then removing the liquid yields a product which can be dyed with acid or metalliferous dyes. The monomers of the polymers used have formula—

$$CH_2 : CH{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}NR^1R^2$$

(R^1 and R^2 = H or the same or different sat. or unsat. aliphatic hydrocarbon radicals or together with the adjacent N atoms form a heterocyclic ring), e.g. a polymeric vinyl ether of diethylaminoethanol. C. O. C.

Coating Paper (I p. 413).
High Polymer Films. V—Effects of Plasticisers, Resins, and Pigments on Viscoelastic Properties of Cellulose Nitrate Films (V p. 422).
Coating Fibres with the help of Gas Agitation (X p. 428).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Heats of Wetting of Collagen, Leather, and other Organic and Fibrous Materials. J. R. Kanagy. *J. Amer. Leather Chem. Assocn.*, **49**, 646–658 (Oct. 1954).

The heats of wetting of collagen, various tanned leathers, and the commoner textile fibres have been measured accurately in a calorimeter. The integral heats have been found to be proportional to the moisture content of the substance at 65% R.H. Nevertheless, there are differences in the initial adsorption energy according to the substance; e.g. wool and vegetable-tanned leather have exceptionally high initial adsorption energies (7050 and 6330 cal./mole of water respectively), while silk has a value of only 2750 cal./mole of water. These differences, and more particularly the difference between the various types of leathers and collagen, are discussed and compared with data calculated from adsorption measurements. W. J. M.

Accessibility of the Cationic Groups of Collagen to large Anions of Polyacids. A. Larsson and K. H. Gustavson. *J. Soc. Leather Trades Chem.*, **39**, 107–123 (April 1955).

In the irreversible fixation by collagen (hide powder) of polyacids with mol.wt. of the order of a few thousand, such as the high-mol.wt. fraction of lignosulphonic acids, measurements of the amounts of protons and anions removed by the protein indicate complete inactivation of the ionic protein groups in the pH range 1·5–2. This method of estimation only proves the complete inactivation of the COO^- ions of collagen. The absence of fixation of protons (0·1 N–HCl) by the inactivated collagen is only an indication of the complete discharge of the COO^- ions of collagen. The degree of irreversible inactivation of the cationic protein groups by the multivalent anions cannot be estimated by these methods. Lignosulphonic acids of average mol.wt. 5000–6000, containing 10–12 SO_3H groups in the anion, show stoichiometric binding of the proton as well as the anion by collagen. To ascertain the number of cationic protein groups of collagen irreversibly inactivated by the anions of lignosulphonic acid, the difference in the heat of reaction of collagen with these acids and HCl has been determined. About half the cationic groups of collagen are thus inactivated; this result is confirmed by an independent method (also described). The remaining cationic groups are probably electrostatically compensated by some long-range effect of the electronegative sites in the large anion of the fixed sulpho acid. J. W. D.

Surface Films of Synthetic Polypeptides. E. Mishuck and F. R. Eirich. *J. Polymer Sci.*, **16**, 397–415 (April 1955).

Three synthetic polypeptides have been spread on aqueous substrates, and surface areas, pressures, and compressibilities measured. On pure water spreading occurs best in the form of condensed films, but on aqueous solutions spreading improves with solute concentration, especially near saturation with lyotropic compounds such as urea or potassium thiocyanate. Molecular weights and surface areas obtained as a function of nature of substrate are related. Conditions and assumptions are discussed on which such a relation can be used to determine true molecular weights. W. R. M.

PATENTS

Deliming Pelts. Riverstone Meat Co. Proprietary. *BP* 728,552

$MgCl_2$, $Mg(NO_3)_2$, Mg acetate, $ZnCl_2$, Zn acetate, $Zn(NO_3)_2$, $AlCl_3$ and $Al(NO_3)_3$ are better deliming agents than NH_4Cl and borax. They are used in aqueous solution at 90–95°F. the pelts being stirred in the liquor for 1 hr. and then rinsed. C. O. C.

Synthetic Tanning Agents. United Shoe Machinery Corpn. *USP* 2,676,870

A polyhydric phenol is treated under acid conditions with an alkyl nitrile and a cyanide. Part of the phenol forms with the nitrile a hydroxy aromatic ketimine and another part combines with HCN liberated from the cyanide to form an aromatic hydroxy aldimine. The ketimine and aldimine then condense together to yield a tanning agent which is believed to be a chalcimine substituted by hydroxyl groups in the aromatic nuclei. Used in the normal manner they convert skins into soft flexible leather of relatively high shrink point. C. O. C.

Synthetic Tannins. Tanexco. *USP* 2,676,980

The protannins (materials usually associated with natural tannin but not normally acting as tanning agents) are extracted from wood by leaching with alkali to produce an extract of pH 6·0–8·5. This is then treated with SO_2 to bring the pH to < 4 and heated under pressure at 150°C. The product may be used for tanning the skins in the normal way to yield a leather of acceptable colour and shrinkage temperature of ca. 75°C. C. O. C.

Filling of Leather. ICI. *BP* 728,771

Modification of *BP* 583,096 (*J.S.D.C.*, **63**, 160 (1947)) better results being obtained by carrying out the tanning in conjunction with drumming in an aqueous dispersion of polyvinylchloride or of a water-insoluble interpolymer of vinylchloride with an ethylenically unsaturated compound, no particle of the polymer being $> 0{\cdot}2\mu$, preferably $> 0{\cdot}1\mu$. C. O. C.

XIII—RUBBER; RESINS; PLASTICS

Surface Chemistry of High Polymers. VI—Monolayer of Polyethylene Terephthalate and Poly-*N*-vinylpyrrolidone. H. Hotta. *Bull. Chem. Soc. Japan*, **28**, 64–67 (Jan. 1955).

From measurements of interfacial pressure and potential of monolayers of polyethylene terephthalate and poly-*N*-vinylpyrrolidone at air–water and oil–water interfaces, it is concluded that rings in the main polymer chain make the polymer molecule rigid. The film of poly-*N*-vinylpyrrolidone is relatively sensitive to alkaline soln.

A. J.

Dye-sensitised Photopolymerisation of Styrene. M. Koizumi, A. Watanabe, and Z. Kuroda. *Nature*, **175**, 770–771 (30 April 1955).

The photopolymerisation of styrene in solution is accelerated by various cyanine dyes. At the same time the dye molecules are decomposed. The presence of dye does not affect the rate of thermal polymerisation. It is suggested that excited dye molecules interact directly with styrene monomer and that the initiation of polymerisation is due to energy transfer. The combination of styrene and zinc tetraphenylchlorin is cited as an example of photosensitised polymerisation due to energy transfer. Initiation is induced by energy transferred from the triplet state of the chlorin compound to the styrene molecule.

W. R. M.

New Chelating Resins. J. R. Parrish. *Chem. and Ind.*, 386–387 (2 April 1955).

New chelating products are described—(*a*) By condensation of H·CHO, resorcinol, and pyrogallolcarboxylic acid; this resin affords a coloured complex with Ba, Ca, and Sr ions in alkaline soln., but, since it only operates at ~ pH 10, analytical use is limited, although Ca can be removed by it from coloured soln. (e.g. molasses) containing an excess of alkali salts. (*b*) By diazotisation of a resin of the *o*-aminophenol type and coupling with 2-naphthol; this resin chelates Cu at low pH and enables the quant. separation of Mg and Cu. (*c*) By condensation of *o*-nitro phenol, resorcinol, and H·CHO followed by reduction; this resin permits the separation of Cu and Ca at ~ pH 5·5. (*d*) By condensation of oxine, resorcinol, and H·CHO; the resulting resin has only low chelating properties. Failures due to excessive cross-linking are avoided by attaching cpd. of the phenolic or amino type to polystyrene resins by the azo linkage. Separations by these chelating resins are rapid because chromatographic elution is unnecessary.

H. H. H.

Furfural in the Synthetic Resin Industry. J. M. a Nolla and E. Márquez. *Afinidad*, 469–476 (Sept.–Oct. 1954).

Uses in modified phenolics and amino and keto resins are reviewed. Formation of resins *in situ* and humectant properties are discussed.

S. R. C.

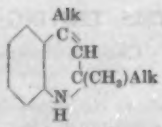
PATENTS

Surface-coated Silicon-containing Particles as Reinforcing Pigments for Light-coloured or White Rubbery Compositions. B. F. Goodrich Co. *BP* 728,227

Silicon monoxide or polymeric silicon oximide of particle size 5–200 mμ., preferably 20–40 mμ., coated with an organosiloxane preferably a polymeric organosiloxane obtained by hydrolysis of an organic halogenosilane, e.g. vinylchlorosilane, is an excellent reinforcing pigment which imparts properties and characteristics, other than colour, normally obtained by use of carbon black. C. O. C.

Rubber and Rubber-like Elastomers having Improved Resistance to Deterioration. Monsanto. *BP* 727,048

A mixture of 6- and 8-alkoxydihydrotrialkylquinolines of formula—



is incorporated in the vulcanisation mixture.

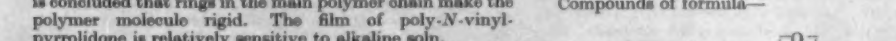
C. O. C.

Stabilisers for Vinyl and Vinylidene Halide Polymers and Copolymers. Monsanto. *BP* 728,029

Cadmium monocyclohexylmaleate or a cadmium mono-(alkylcyclohexyl)maleate is a good heat stabiliser preventing both discoloration and in the case of thin films formation of haze. C. O. C.

Epoxy Acyl Ricinoleates as Plasticisers for Vinyl Polymers and Copolymers. Monsanto. *BP* 728,258

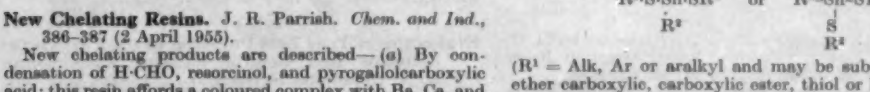
Compounds of formula—



(R^1 and R^2 = Alk of 1–5 C; T = alkylene of 2–6 C, alkyleneoxyalkylene of 4–8 C or polyalkyleneoxyalkylene of 6–10 C), e.g. 2′-(2″-acetoxyethoxy)ethyl 9:10-epoxy-12-acetoxyoctadecanoate, when used as plasticisers for vinyl polymers or copolymers yield compositions of good thermal stability. They do not react with the commercially available heat and light stabilisers. C. O. C.

Organotin Compounds as Stabilisers for Vinyl Chloride Polymers and Copolymers. Firestone Tire & Rubber Co. *BP* 728,953–4

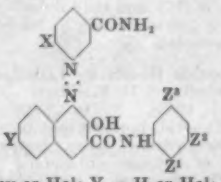
Compounds of formulae—



(R^1 = Alk, Ar or aralkyl and may be substituted by an ether carboxylic, carboxylic ester, thiol or hydroxy group or by Br, Cl or I on an aromatic ring C atom or by F on any C atom or by an aliphatic hydrocarbon radical containing an ethylenic or acetylenic linkage or R^1 = a heterocyclic radical attached to the S atom by a C atom; R^2 = Alk, Ar or heterocyclic radical attached to the Sn atom by a C atom and may be substituted by an ether radical), e.g. di-*n*-butyltin diphenyldimercaptide, impart to vinylchloride polymers outstanding resistance to heat and mechanical working. C. O. C.

Colouring Vinyl Polymers Red. FH. *BP* 728,601

Incorporation into vinyl polymers of a water-insoluble dye of formula—



(X = Alk, alkoxy or Hal; Y = H or Hal; Z^1 = methoxy; Z^2 = methoxy when Z^3 = methyl, methoxy or Hal and *vice versa*), e.g. 1-amino-2-methylbenzene-5-carboxylic acid amide → 1-(2′:3′-hydroxynaphthoylamino)-2:5-dimethoxy-4-chlorobenzene, colours them red of very good fastness, the dye being inert to the plasticisers commonly used. Vinyl polymers so coloured neither bloom nor bleed into other vinyl polymers with which they may be in contact. C. O. C.

Continuous Production of Sheets or Tubes of Thermoplastic Materials having Stripes of Two or More Colours. Plastar S/A Comercio e Industria de Materiais e Produtos Plasticos. *BP* 728,025

Dyeing Plastic Sheets for Use in Glare Screens (I p. 413).
Pigmenting Synthetic Linear Polymers before Melt Spinning or Extrusion (VIII p. 425).

XIV—ANALYSIS; TESTING; APPARATUS

Applications of Chromatography in Inorganic Analysis. J. M. Piera. *Afinidad*, 492–496 (Sept.–Oct. 1954).

Reference is made to the separation of metal ions on prepared alumina, a mixture of 1 part 8-hydroxyquinoline to 2 parts kieselguhr and paper adsorbents. Two-dimensional chromatography was also employed. S. R. C.

Application of the Catalytic Decomposition of Hydrogen Peroxide at a Platinum Electrode in Potentiometric Titration. I. E. Flis. *J. Anal. Chem. U.S.S.R.*, **10**, 38–43 (Jan.–Feb. 1955).

The potential of a Pt electrode in a soln. containing a little H_2O_2 is a linear function of the pH (it is about 0·4 v. less than the potential of an oxygen electrode). A potentiometric curve for the titration of a soln. containing $NaClO_2$, NaOH, and Na_2CO_3 with acid with the aid of this electrode is identical with that obtained at a glass electrode. The complete analysis of a soln. containing NaOCl, NaOH, and Na_2CO_3 is performed by combining the results of two potentiometric titrations—direct titration with acid at a Pt electrode, which acts essentially as an oxidation–reduction electrode, and a similar titration performed after addition of excess of H_2O_2 to decompose the hypochlorite and to convert the Pt electrode into a pH electrode for the non-oxidative system. A. E. S.

Specific Reaction for Formaldehyde. G. Vanag and E. Vanag. *J. Anal. Chem. U.S.S.R.*, **10**, 63–64 (Jan.–Feb. 1955).

The soln. to be tested (1–2 drops) is added to 2-nitro-indan-1:3-dione (5–10 mg.). After 15 min. conc. H_2SO_4 (1 ml.) is added, and the mixture is warmed. In presence of formaldehyde the soln. acquires a green fluorescence. The reaction is highly specific, and 3 μg. of HCHO in 0·1 ml. of soln. can be detected. A. E. S.

Detection of Casein in Paper. R. G. Knapman and I. O. Wilson. *Proc. Tech. Sectn. British Paper & Board Makers' Assocn.*, **35**, 537–540 (Dec. 1954).

As the red colour produced with Millon's reagent is not specific for tyrosine, but can be obtained with certain other phenolic compounds, the test results may be misinterpreted through the presence of a phenolic preservative. An alternative procedure, based on Hehner's method for detecting formaldehyde, is thought to be more reliable and has been shown to be sensitive to ca. 0·5% of casein in paper. S. V. S.

Estimation of Sulphur in Proteins, particularly in Leather. P. H. Rao. *J. Amer. Leather Chem. Assocn.*, **49**, 755–757 (Dec. 1954).

The material is oxidised by ignition with Na_2O_2, the ash is taken up in HCl, and the sulphate is pptd. by $BaCl_2$ and determined gravimetrically. The method is quick, reliable, and accurate. J. W. D.

Fission of Peptide Bonds by Lithium Borohydride. J. C. Crawhall and D. F. Elliott. *Nature*, **175**, 299–300 (12 Feb. 1955).

The fission of peptide linkages may be effected by means of lithium borohydride, those bonds involving the COOH group of glycine or alanine being especially susceptible. The cleavage of small mol. such as di- or tri-peptides proceeds satisfactorily at room temp., though larger mol. may require heat and agitation. It thus appears that $LiBH_4$ is not reliable as an end-group reagent for large peptides or proteins, since there is no differentiation between the end-groups initially present and those produced by chain-fission. J. W. D.

PATENT

Instrument for Testing Flexible Sheet Material. Johnson & Johnson. *BP* 729,106

A sheet of flexible material is placed on a platform which has a slot extending widthwise of the paper, and a blade is mounted on the end of a counterpoised lever in such a manner that when it moves downwards it pushes the sheet through the slot in the form of a U-shaped loop. The force required to do this is a measure of the flexibility of the sheet, and is transmitted to a strain gauge situated near the fulcrum. This enables it to be measured electrically, by way of a microammeter reading. The cycle in which the beam tilts, pushes the material, and returns to its original position is performed automatically. J. W. B.

Photodegradation of Cellulose, particularly in the Presence of Titanium Dioxide (VI p. 422).

Insolubility of Cuprammonium Hydroxide as a Means of detecting Cross-linking in chemically Modified Cotton (X p. 427).

Antistatic Action and Molecular Structure (X p. 427).

XV—MISCELLANEOUS

The Twelve Whites. E. N. Willmer. *Endeavour*, **14**, 19–23 (Jan. 1955).

The author describes in detail twelve different methods whereby the eye can receive the impression of white. Some of these methods are physical and are produced by well known means. Others depend upon principles which, although well recognised, are not understood. The paper is excellently illustrated with coloured plates. W. J. M.

The Colours of Animals. H. M. Fox. *Endeavour*, **14**, 40–47 (Jan. 1955).

The colours of animals are discussed and illustrated with some excellent photographs. These colours are due either to pigments or to physical structure, sometimes modified by pigments. An important group of pigments, the carotenoids, are derived from plant foods, but many others are synthesised by the animals. Structural colours are produced by Tyndall scattering or by interference, but rarely by diffraction, and in some cases these colours are modified by the presence of pigments. The function of animal coloration is briefly discussed, and while much of this is for some definite purpose, a few examples are quoted, such as the beautiful colours in mother of pearl, in which the colour can have no practical value. W. J. M.

Evaluation of True Zeta Potential of the Particles of Cellulose and Gelatin forming Diaphragms. B. N. Ghosh and S. Ghosh. *J. Indian Chem. Soc.*, **31**, 649–653 (Sept. 1954).

It is shown from the data on streaming potential measurements with oxycelluloses of Rabinov and Heymann (*J. Phys. Chem.*, **47**, 655 (1943)) and with filter papers (Briggs, *ibid.*, **32**, 641 (1928)) that the true ζ potential can be calculated from the expression—

$$\frac{\zeta}{\zeta_a} = 1 + \frac{a}{S}(K_p - S)$$

The true ζ potential of a gelatin gel at pH 3·5 can be evaluated from the expression—

$$\frac{\zeta}{\zeta_a} = 1 + \frac{a}{S} Kyn$$

(ζ_a = electrokinetic potential calculated from Smoluchowski's equation; ζ = true ζ potential; a = correction factor > 1; S = specific bulk conductivity of soln.; K_p = specific conductivity of soln. inside the diaphragm; n is proportional to the concn. of gelatin; and K and y are constants for the experimental condition. A. J.

Glass coloured by Colloids—I. S. Kawakubo. *J. Ceram. Assocn. Japan*, **62**, 565–568 (1954): *Chem. Abs.*, **49**, 2689 (25 Feb. 1955).

Study of Se ruby glass containing no S or Zn shows that the Se ruby colour can be produced by adding Cd selenide or a mixture of Cd selenite and a reducing agent, e.g. Al or Si powder, directly to a batch of ordinary soda–lime glass. With the selenite the ruby colour appears only when the stoichiometric relations are satisfied between the amount of Cd selenite and the reducing agent. Apparently presence of Cd selenide is necessary for production of Se ruby glass, whereas presence of sulphide sulphur and Zn is unnecessary. C. O. C.

PATENTS

Stain-Filler-Sealer for Wood. Chadeloid Corpn. *USP* 2,674,538

A mixture of shellac (which must be free from wax and rosin), an alcohol, a natural gum, a dye and an inert filler, e.g. silica, asbestine or talc, is used. C. O. C.

This reproduction of an actual print on delicate nylon illustrates one interesting use.
Though Cotton and Spun rayon provide by far the greatest outlet, ARIDYE pigment colours are successfully applied to almost every type of fibre, both natural and synthetic.

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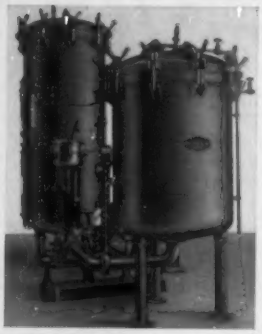

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All inquiries relating to Advertisements in the Journal should be addressed to THE GENERAL SECRETARY, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

Replies may be addressed Box —, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

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The engagement of persons answering these advertisements must be made through a Local Office of the Ministry of Labour or a Scheduled Employment Agency if the applicant is a man aged 18–64 inclusive or a woman aged 18–59 inclusive unless he or she, or the employment, is excepted from the provisions of the Notification of Vacancies Order 1952.

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Persons desirous of joining the Society as Ordinary Members or Junior Members may obtain Application Forms from the Secretary, or from the Honorary Secretary of any Section of the Society.

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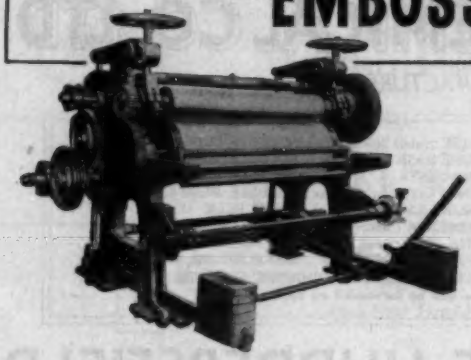

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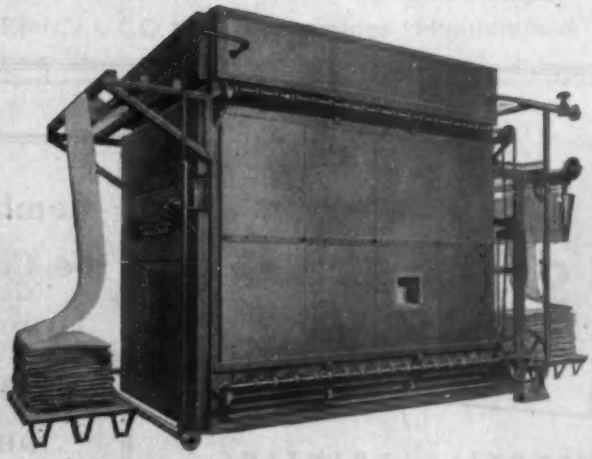

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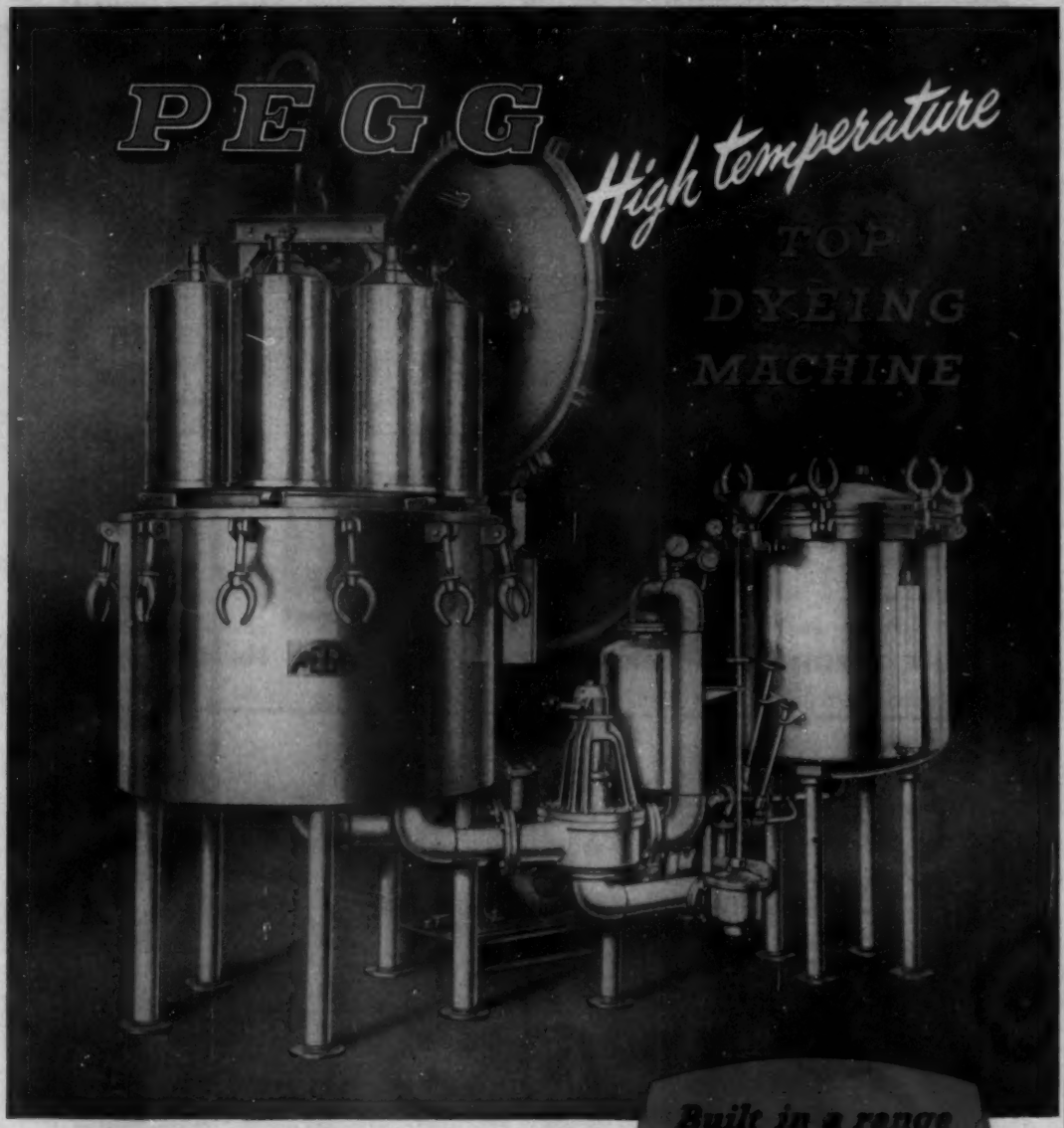

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